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Guidelines for the quality assurance of the determination of beryllium emission rates from stationary sources by the Federal reference method are presented. These include:

1. Good operating practices
2. Directions on how to assess performance and qualify data
3. Directions on how to identify trouble and improve data quality
4. Directions to permit design of auditing activities

The document is not a research report. It is designed for use by operating personnel.

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by

P. S. Wohlschlegel
F. Smith
D. E. Wagoner

Research Triangle Institute
Research Triangle Park, North Carolina 27709

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Environmental Monitoring and Support Laboratory
Office of Monitoring and Technical Support
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April 1976

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TABLE OF CONTENTS

Section		Page
	LIST OF FIGURES	v
	LIST OF TABLES	vi
	ABSTRACT	vii
I	INTRODUCTION	1
II	OPERATIONS MANUAL	5
	2.0 GENERAL	6
	2.1 APPARATUS SELECTION	10
	2.2 EQUIPMENT CALIBRATION	19
	2.3 PRESAMPLING PREPARATION	27
	2.4 ON-SITE MEASUREMENTS	41
	2.5 POSTSAMPLING OPERATIONS (Base Laboratory)	55
III	MANUAL FOR FIELD TEAM SUPERVISOR	69
	3.0 GENERAL	70
	3.1 ASSESSMENT OF DATA	71
	3.2 SUGGESTED PERFORMANCE CRITERIA	74
	3.3 COLLECTION AND ANALYSIS OF INFORMATION TO IDENTIFY TROUBLE	74
IV	MANUAL FOR MANAGER OF GROUPS OF FIELD TEAMS	89
	4.0 GENERAL	90
	4.1 FUNCTIONAL ANALYSIS OF TEST METHOD	93
	4.2 ACTION OPTIONS	102
	4.3 PROCEDURES FOR PERFORMING A QUALITY AUDIT	107
	4.4 DATA QUALITY ASSESSMENT	115
	LIST OF REFERENCES	128

TABLE OF CONTENTS (Continued)

Section Page

APPENDIXES

APPENDIX A	REFERENCE METHOD	132
APPENDIX B	ILLUSTRATED USE OF NOMOGRAPHS	138
APPENDIX C	ILLUSTRATED AUDIT PROCEDURES	144
APPENDIX D	GLOSSARY OF SYMBOLS	150
APPENDIX E	GLOSSARY OF TERMS	153
APPENDIX F	CONVERSION FACTORS	155

LIST OF FIGURES

Figure No.	Title	Page
1.	Operational flow chart of the measurement process.	7-9
2.	Sampling train.	11
3.	Dry gas meter and orifice meter calibration and calculation form.	23
4.	Beryllium measurement checklist.	30-32
5.	Sample data form for beryllium emissions determinations.	42-44
6.	Sample calculation and data analysis form.	62-63
7.	Sample control for the range, RG, of R replicates.	84
8.	Sample control chart for the range, RG, of percent isokinetic, I, sampling for three test runs per field test.	85
9.	Sample control chart for the average percent of isokinetic sampling per field test.	86
10.	Sample control chart for the measurement of working standard solutions.	88
11.	Summary of data quality assurance program.	94
12.	Added cost vs precision for selected action options for mercury emission rate determinations.	106
13.	Average cost vs audit level (n).	109
14.	Sample form for recording audit data.	113
15.	Example illustrating $p < 0.10$ and satisfactory data quality.	121
16.	Example illustrating $p > 0.10$ and unsatisfactory data quality.	121
17.	Flow chart of the audit level selection process.	124

LIST OF TABLES

Table No.	Title	Page
1.	Suggested Performance Criteria.	75
2.	Factors for Control Charts for the Range, RC.	83
3.	Means and Variabilities of Parameters Affecting V_{total}.	102
4.	Beryllium Emission Determination Checklist to be Used by Auditor.	110-111
5.	Computation of the Mean Difference, $\bar{d}$, and Standard Deviation of the Differences, s_d.	118
6.	Sample Plan Constants, k for P{Not Detecting a Lot with Proportion P outside Limits L and U} $\leq$ 0.1.	122

ABSTRACT

Guidelines for the quality assurance of the determination of beryllium emission rates from stationary sources by the Federal reference method are presented. These include:

1. Good operating practices
2. Directions on how to assess performance and qualify data
3. Directions on how to identify trouble and improve data quality
4. Directions to permit design of auditing activities

The document is not a research report. It is designed for use by operating personnel.

This work was submitted in partial fulfillment of Contract Durham 68-02-1234 by Research Triangle Institute under the sponsorship of the Environmental Protection Agency. Work was completed as of February 1976.

SECTION I

INTRODUCTION

SECTION I INTRODUCTION

This document presents guidelines for developing a quality assurance program for Method 104, Determination of Beryllium Emissions from Stationary Sources. This method was published by the Environmental Protection Agency in the Federal Register, April 6, 1973, and is reproduced as appendix A of this report for convenience of reference.

This document is divided into four sections:

Section I, Introduction. The introduction lists the overall objectives of a quality assurance program and delineates the program components necessary to accomplish the given objectives.

Section II, Operations Manual. This manual sets forth recommended operating procedures to insure the collection of data of high quality, and instructions for performing quality control checks designed to give an indication or warning that invalid data or data of poor quality are being collected, allowing for corrective action to be taken before future measurements are made.

Section III, Manual for Field Team Supervisor. This manual contains directions for assessing data quality on an intralaboratory basis and for collecting the information necessary to detect and/or identify trouble.

Section IV, Manual for Manager of Groups of Field Teams. This manual presents information relative to the test method (a functional analysis to identify the important operations variables and factors, and statistical properties of and procedures for carrying out auditing procedures for an independent assessment of data quality).

The objectives of this quality assurance program for Method 104 are to:

1. Minimize systematic errors (biases) and control random variability (precision) within acceptable limits in the measurement process,
2. Provide routine indications for operating purposes of satisfactory performance of personnel and/or equipment,
3. Provide for prompt detection and correction of conditions that contribute to the collection of poor quality data, and
4. Collect and supply information necessary to describe the quality of the data.

To accomplish the above objectives, a quality assurance program must contain the following components:

1. Recommended operating procedures,
2. Routine training of personnel and evaluation of performance of personnel and equipment,
3. Routine monitoring of the variables and parameters that may have a significant effect on data quality,
4. Development of statements and evidence to qualify data and detect defects, and
5. Action strategies to increase the level of precision/accuracy in the reported data.

Component (2) above will be treated for all the methods in the final report of this contract.

Implementation of a properly designed quality assurance program should enable measurement teams to achieve and maintain an acceptable level of precision and accuracy in their stack gas composition measurements. It will also allow a team to report an estimate of the precision of its measurements for each source emissions test.

Variability in emission data derived from multiple tests conducted at different times includes components of variation from:

1. Process conditions,
2. Equipment and personnel variation in field procedures, and
3. Equipment and personnel variation in the laboratory.

In many instances time variations in source output may be the most significant factor in the total variability. The error resulting from this component of variation is minimized by knowing the time characteristics of the source output and collecting the gas sample at a rate proportional to the stack gas velocity. The sampling period should span at least one complete output cycle when possible. If the cycle is too long, either the sample collection should be made during a portion of the cycle representtative of the cycle average, or multiple samples should be collected and averaged.

Quality assurance guidelines for Method 104 as presented here are designed to insure the collection of data of acceptable quality by prevention, detection, and quantification of equipment and personnel variations in both the field and the laboratory through:

1. Recommended operating procedures as a preventive measure,

2. Quality control checks for rapid detection of undesirable performance, and
3. A quality audit to independently verify the quality of the data.

The scope of this document has been purposely limited to that of a field and laboratory document. Additional background information is contained in the final report under this contract.

2.0 GENERAL

This manual sets forth recommended procedures for the determination of beryllium (Be) emissions from stationary sources according to Method 104 (Method 104 is reproduced from the Federal Register and is included as appendix A of this document). Quality control procedures and checks designed to give an indication or warning that invalid or poor quality data are being collected are written as part of the operating procedures and are to be performed by the operator on a routine basis. In addition, the performance of special quality control procedures and/or checks as prescribed by the supervisor for assurance of data quality may be required of the operator on special occasions.

The sequence of operations to be performed for the measurement process is given in figure 1. Each operation or step in the method is identified by a block. Quality checkpoints in the measurement process, for which appropriate quality control limits are assigned, are represented by blocks enclosed by heavy lines. Other quality checkpoints involve go/no-go checks and/or subjective judgments by the test team members with proper guidelines for decisionmaking spelled out in the procedures.

The precision/accuracy of data obtained from this method depends upon equipment performance and the proficiency with which the operator performs his various tasks. From equipment calibration through on-site measurement, calculations, and data presentation, this method is susceptible to a variety of errors. Detailed instructions are given for minimizing or controlling equipment error, and procedures designed to minimize personnel errors are recommended. Before using this document, the operator should study Method 104 as reproduced in appendix A in detail. In addition, the quality assurance documents of this series for Methods 2, 3, and 4 should be read and followed.

It is assumed that all apparatus satisfies the reference method specifications and that the manufacturer's recommendations will be followed when using a particular piece of equipment. It is also assumed that the analyst performing the analyses is trained in the operation of an atomic absorption spectrometer utilizing a N_2O/acetylene burner.

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APPARATUS SELECTION

1. SELECT EQUIPMENT ACCORDING TO THE GUIDELINES GIVEN IN SUBSECTION 2.1 FOR THE SOURCE TO BE TESTED.

EQUIPMENT CALIBRATION

2. CALIBRATE EQUIPMENT ACCORDING TO SUBSECTION 2.2.

PRESAMPLING PREPARATION

3. OBTAIN PROCESS DATA, SELECT/PREPARE SAMPLING SITE, DETERMINE LOGISTICS FOR PLACING EQUIPMENT ON-SITE, and DETERMINE STACK CONDITIONS T_s, V_s, B_{wo}, AND M_d.

4. CHECK OUT SAMPLING TRAIN AND RELATED COMPONENTS.

5. PACKAGE AND SHIP EQUIPMENT.

ON-SITE MEASUREMENTS

6. MOVEMENT OF EQUIPMENT TO SAMPLING SITE AND SAMPLE RECOVERY AREA.

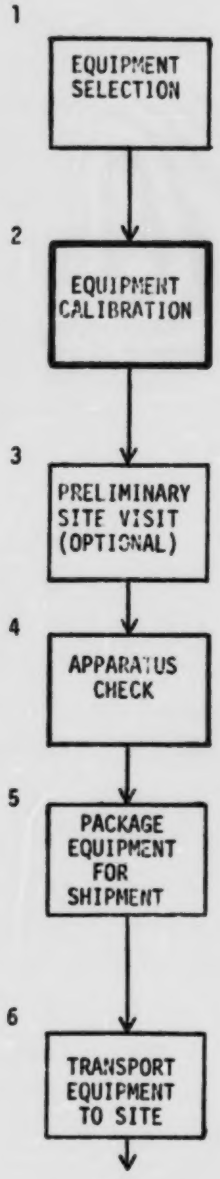
1
EQUIPMENT SELECTION
2
EQUIPMENT CALIBRATION
3
PRELIMINARY SITE VISIT (OPTIONAL)
4
APPARATUS CHECK
5
PACKAGE EQUIPMENT FOR SHIPMENT
6
TRANSPORT EQUIPMENT TO SITE

Figure 1. Operational flow chart of the measurement process.

7. PRELIMINARY MEASUREMENTS AND SETUP WILL INCLUDE DUCT MEASUREMENT, PERCENT H_2O, MOLECULAR WEIGHT (M_s), AND SAMPLE BOX LOGISTICS.

8. DETERMINATION OF MAXIMUM AND MINIMUM ΔP AND STACK GAS TEMPERATURE.

9. SET NOMOGRAPH UTILIZING THE PRELIMINARY STACK GAS PARAMETERS [app. B].

10. ASSEMBLE AND LEAK-CHECK THE SAMPLING TRAIN.

11. COLLECT A MINIMUM SAMPLE VOLUME OF $1.7\ m^3$ ($60\ FT^3$). MAINTAIN ISOKINETIC CONDITIONS DURING SAMPLING.

12. PERFORM FINAL LEAK CHECK OF THE ENTIRE SAMPLING TRAIN.

13. OBTAIN SAMPLE FROM FILTER HOLDER, PROBE NOZZLE AND IMPINGERS. MEASURE TOTAL CONDENSATE (IMPINGER VOLUME AND SILICA GEL WEIGHT GAIN) ACETONE, WATER WASHES.

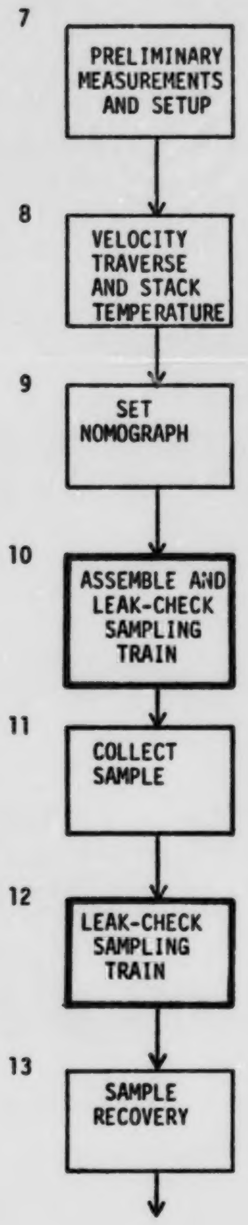
7
PRELIMINARY MEASUREMENTS AND SETUP
8
VELOCITY TRAVERSE AND STACK TEMPERATURE
9
SET NOMOGRAPH
10
ASSEMBLE AND LEAK-CHECK SAMPLING TRAIN
11
COLLECT SAMPLE
12
LEAK-CHECK SAMPLING TRAIN
13
SAMPLE RECOVERY

Figure 1. Operational flow chart of the measurement process--continued.

14. VALIDATE THE DATA BY CHECKING ALL DATA SHEETS FOR COMPLETENESS.

15. DISASSEMBLE AND INSPECT EQUIPMENT FOR DAMAGE SUSTAINED BUT NOT DETECTED DURING SAMPLING.

16. PACKAGE EQUIPMENT FOR RETURN TRIP TO BASE LABORATORY.

POSTSAMPLING OPERATIONS

17. PREPARE SAMPLES FOR ANALYSIS.

18. PREPARE WORKING ANALYTICAL CURVE AND ANALYZE SAMPLES.

19. PERFORM NECESSARY CALCULATIONS TO OBTAIN RATE OF BERYLLIUM EMISSIONS AND PERCENT ISOKINETIC VARIATION.

20. FORWARD THE DATA FOR FURTHER INTERNAL REVIEW OR TO THE USER.

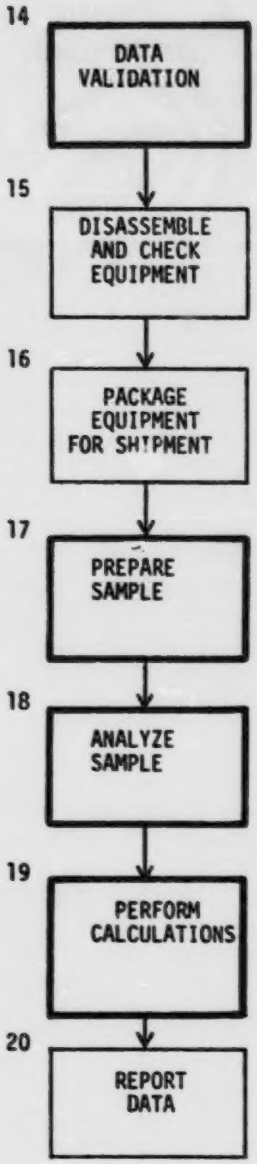
14
DATA VALIDATION
15
DISASSEMBLE AND CHECK EQUIPMENT
16
PACKAGE EQUIPMENT FOR SHIPMENT
17
PREPARE SAMPLE
18
ANALYZE SAMPLE
19
PERFORM CALCULATIONS
20
REPORT DATA

Figure 1. Operational flow chart of the measurement process--continued.

2.1 APPARATUS SELECTION

A schematic of an assembled beryllium sampling train with all components identified is shown in figure 2. Additional specifications, criteria and/or design features as applicable are given in this section to aid in the selection of equipment to insure the collection of data of consistent quality. Procedures and, where applicable, limits for acceptance checks are given. The descriptive title, identification number, if applicable, and the results of the acceptance check are recorded in the receiving record file, dated, and signed by the individual performing the check.

2.1.1 Nozzle

2.1.1.1 Design Characteristics. Design of the nozzle, serving as an interface between the sampling probe and the flue gas, should result in minimum disturbance of the flow pattern and sample gas characteristics. The nozzle must be inert to the sample gases at the temperatures encountered in the field. A new nozzle should be visually checked for identification, i.e., verify that it is the size, shape, and composition ordered. The nozzle should also be checked for damage according to subsection 2.3.2.1(1), and the nozzle tip diameter should be measured according to subsection 2.2.1. A nozzle not satisfying any one of the above checks should be rejected.

The nozzle should be thin-walled with a beveled, sharp leading edge. The bevel should be on the outside of the nozzle with a continuous, smooth inside surface, i.e., a constant internal diameter must be preserved. A button-hook-shaped nozzle (see figure 2) is required to allow for easy insertion through small ports when sampling a thick-walled stack. For nozzles greater than 1.90 cm (3/4 in) inside diameter, either an L-shaped or a smooth, continuous 90-degree-bend nozzle is acceptable.

A set of three nozzles with 6.4 mm (1/4 in.), 9.5 mm (3/8 in.) and 13 mm (1/2 in.) inside diameters is recommended. Low stack velocities, high moisture content, or high stack temperatures may require 5 mm (3/16 in.) or 15 mm (5/8 in.) diameter nozzles in order to achieve isokinetic sampling. These larger sizes are not stock items but are available from the manufacturer by special order.

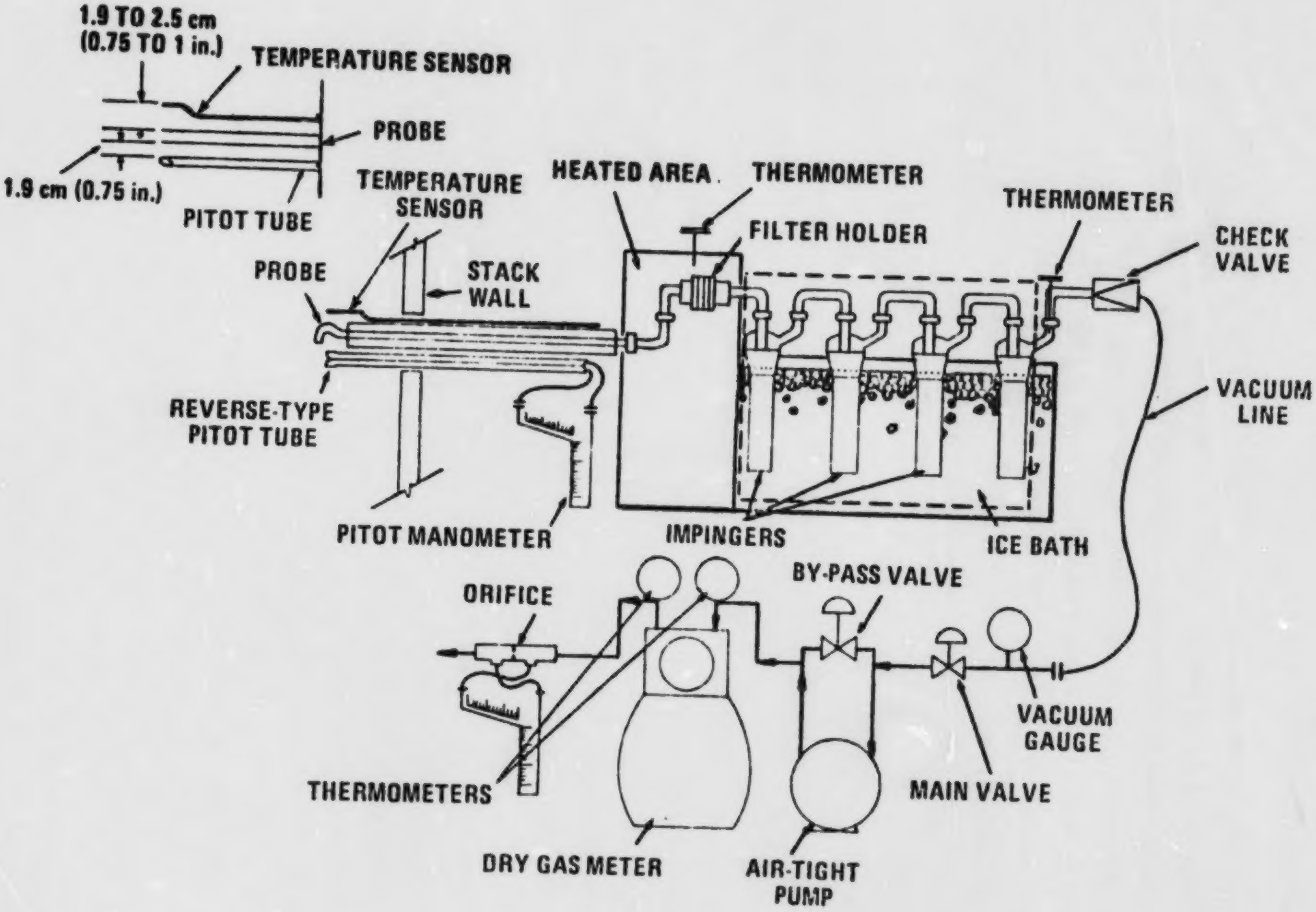
1.9 TO 2.5 cm
(0.75 TO 1 in.)
TEMPERATURE SENSOR
PROBE
1.9 cm (0.75 in.)
PITOT TUBE
TEMPERATURE SENSOR
PROBE
STACK WALL
REVERSE-TYPE PITOT TUBE
PITOT MANOMETER
HEATED AREA
THERMOMETER
FILTER HOLDER
THERMOMETER
CHECK VALVE
VACUUM LINE
IMPINGERS
ICE BATH
ORIFICE
BY-PASS VALVE
VACUUM GAUGE
MAIN VALVE
THERMOMETERS
DRY GAS METER
AIR-TIGHT PUMP

Figure 2. Beryllium sampling train.

The following specifications are recommended:

Material:	
Temperatures below 871°C (1600°F)	Seamless stainless steel (316) tubing for 2- to 3-hour exposures (ref. 1).
Temperatures above 871°C (1600°F)	Quartz
Temperatures up to 1371°C (2500°F)	Nickel-base alloys such as Inconel (subject to severe corrosion in the presence of fluorine or chlorine)
Wall Thickness:	1.65 mm (0.065 in.)
Angle of Taper:	$\leq$ 30°
Distance from Tip of Nozzle to First Bend:	At least two times the outside nozzle diameter
Inside Diameter:	Available in increments of 1.6 mm (1/16 in.)
Probe Nozzle Brush:	See section 2.1.2.

2.1.2 Sampling Probe

The sampling probe should be made of borosilicate (Pyrex) glass and encased in a steel sheath. The probe must be equipped with a heating system which will regulate the probe inlet and exit temperatures to avoid condensation and reevaporation of the moisture in the sample gas. The probe material must be non-reactive with the gas constituents so as not to introduce a bias into the analytical method. Because of this, a knowledge of the stack gas composition and temperature is necessary to select the correct probe. A new probe should be visually checked for identification, i.e., verify that it is the length and composition ordered. The probe should be checked for cracks or breaks and leak-checked on a sampling train as described in subsection 2.3.2.1(2). Also, the probe heating system should be calibrated according to subsection 2.2.2. Any probe not satisfying the acceptance check should be repaired, if possible or rejected.

A probe brush of an appropriate size and shape is necessary to clean the probe liner and nozzle prior to and after sampling.

The following specifications are suggested for the probe and probe brush:

Probe Material:	
Temperature ≤ 480°C (900°F)	Thick-walled borosilicate glass or quartz glass
Temperature ≤ 900°C (1650°F)	Quartz
Inside Diameter:	1.3 cm (.5 in.) approximately
Heating System:	
Temperature requirements at the exit and within .03 m (12 in) of the inlet	No greater than 93 ± 14°C (200 + 25°F) with a gas flow of 0.021 m^3/min (.75 ft^3/min) at room temperature

Note 1: The general requirement here is to prevent condensation prior to the filter during sampling. In most cases a heating capacity to attain and hold 93°C (200°F) at the exit end of the probe under field conditions is sufficient. This general requirement is tempered with the fact that a membrane filter will collapse at ≈ 107°C (225°F).

Probe Brush:	
Bristles	Nylon
Handles	Stainless steel wire
Extensions	Inert material and at least as long as the probe.

2.1.3 Filter Holder

The filter holder should be durable, easy to load, and leak-free in normal applications. The design must be such that the filter material is not torn as the holder is tightened. Also, the only flow through the holder must be through the filter. New filter holders are checked visually for cracks and sharp edges that could puncture or tear a filter. The filter holder with a filter installed is placed in a sampling train and leak-checked at about 380 mm Hg(15 in. Hg) vacuum as directed in subsection 2.4.3.5. Disassemble the holder and check the filter for punctures and cuts. Reject the holder if any of the checks are negative and cannot be corrected.

The glass frit should always be cleaned before sampling according to the manufacturer's recommendations. A standard cleaning procedure is not applicable to all filter supports due to the variation in construction materials, but this construction material should allow the rinsing of the interior surface of the filter holder and frit with 1:1 (V/V) hydrochloric acid and water. This wash should be followed with a final rinse of distilled, deionized water.

Specifications for the filter holder are:

Material:	Borosilicate (Pyrex) glass (other materials may be used if approved by the EPA administration)
Leak Sealant:	Non-reactive to the stack gases
Filter Support Media:	Glass frit

2.1.4 Filter Holder Box

The filter box must be equipped with a heating system and a temperature monitoring device to regulate the temperature around the filter holder during sampling if condensation is a problem. A dial-type thermometer is recommended for monitoring the temperature.

Visually check the filter holder box for damage. Check the heating system by calibrating it as directed in subsection 2.2.2. Reject the box if it is damaged or if a temperature of 93 ± 14°C (200 ± 25°F) cannot be maintained. The heating system and thermometer should meet the following criteria:

Desired Temperature:	At or above stack gas temperature but not greater than 107°C (225°F)
Thermometer:	
Accuracy	± 3°C (± 5.4°F)
Range	16° to 149°C (60° to 300°F)

2.1.5 Impingers/Condenser/Container

The sample gas, having passed through the filter, must be cooled to 20°C (68°F) or less, and the moisture content of the stack gas measured. To accomplish this task, four impingers are connected in series. The first, third and fourth impingers (see figure 2) are of the modified Greenburg-Smith design. The second impinger is of the Greenburg-Smith design with the standard tip. Each impinger is checked visually for damage, such as breaks or cracks, and manufacturing flaws, such as poorly shaped connections. The container should be checked for damage and filled with water to see if it leaks. Reject any item that is faulty.

The fourth impinger is required (charged with 200 g of preweighed silica gel) to remove moisture and to protect the vacuum pump and dry gas meter. The reference method requires that the condensate trap be used to keep the effluent gas temperature at 20^{o}C (68^{o}F) or less. Ice containers are available in commercial trains or can be fabricated from closed-pore expanded polyethylene. The efficiency of the ice bath can be increased by the addition of salt.

Specifications are as follows:

Second Impinger:	Tip must be large enough to allow an impinger full of water to drain in at least 6 to 8 seconds (this avoids excessive pressure drop in the sampling system)
Modified Impingers:	Replace tip with a 13 mm (1/2 in.) diameter glass tube extending to within 13 mm from the bottom of the flask
Joints:	Inert, leak-free

2.1.6 Vacuum Pump

The vacuum pump must be capable of maintaining a constant flow rate of the sample gas. Two types of vacuum pumps are commonly used: a modified sliding fiber vane pump and a diaphragm pump. The pump should be leak-checked upon placing the pump in a sampling train such as in figure 2.

With the pump running, adjust the system to 381 mm Hg (15 in. Hg) with the inlet of the metering system closed. The dry gas meter dial should not move after the adjustment has been made. The absence of valve float must also be verified when a diaphragm pump and by-pass valve are used in the sampling train.

Suggested specifications for the vacuum pump follow:

Desired Flow Rate:	
At zero vacuum	.11 m^3/min (4 ft^3/min)
At 508 mm Hg (20 in. Hg)	.03 m^3/min (1 ft^3/min) (with the pump outlet at or near standard pressure, i.e., 760 mm Hg)
Vacuum (Inlet Plugged):	380 mm Hg (15 in. Hg)

2.1.7 Dry Gas Meter

A new dry gas meter must be calibrated according to section 2.2.3. It should meet the following requirement:

Accuracy:	±2 percent (compared to a wet test meter) at flow rates of .008 to .034 m^3/min (0.3 to 1.2 ft^3/min)

2.1.8 Orifice Meter

Construction details of the orifice meter are given by Martin (ref. 2). Significant design criteria are listed below. After visually inspecting the orifice meter and the inclined manometer (or equivalent differential pressure gage) for damage, the instruments are assembled in a sampling train as shown in figure 2 and calibrated as directed in subsection 2.2.3. If $\Delta H@$ is outside the limits outlined below (and discussed in subsection 2.2.3), the orifice meter should not be used with a commercially available sampling nomograph. If $\Delta H@$ is greater than 53 mm (2.1 in.) of water, the orifice opening can be made larger (ref. 2) to lower $\Delta H@$ to an acceptable value. Low values of $\Delta H@$ cannot be corrected. Values of $\Delta H@$

outside the above limits are acceptable when actual values are used to calculate the relationship of ΔH to ΔP (i.e., a nomograph is not used) for each field test. The value of $\Delta H@$ should be inscribed on or otherwise affixed to the orifice meter box.

Specifications for this instrumentation include:

Orifice Diameter:	47 cm (3/16 in.)
$\Delta H@$ at 21°C (70°F) and 760 mm Hg (29.92 in. Hg):	46.7 ± 6.4 mm H_2O (1.84 ± 0.25 in. H_2O)
Inclined Manometer Range:	0 to 203 or 250 mm H_2O (0 to 8 or 10 in. H_2O)
Inclined Portion of Scale:	
Range	0 to 25 mm H_2O (0 to 1 in. H_2O)
Divisions	0.25 mm H_2O (0.01 in. H_2O)
Vertical Portion of Scale:	
Range	25 to 203 or 254 mm H_2O (1 to 8 or 10 in. H_2O)
Divisions	2.5 mm H_2O (0.1 in. H_2O)

2.1.9 Thermometers

Three dial-type thermometers are suitable for monitoring the inlet and outlet temperatures of the dry gas meter and the sample gas as it leaves the last impinger. All of the above temperatures can be monitored by other means, such as a thermocouple or thermistor, but even then a dial-type thermometer is recommended as a backup system.

Dial-type thermometers are easily damaged. Each new thermometer should be visually checked for damage, such as a dented or bent stem. Each thermometer is then calibrated as directed in subsection 2.2.4. A thermometer should read within ± 2.8°C (± 5°F) of the true value when checked in an ice water bath and/or ± 3.9°C (± 7°F) when checked in boiling water. Damaged thermometers that cannot be calibrated should be rejected.

Suggested thermometer data includes:

Range:	0 to 50°C (30° to 120°F)
Accuracy:	±1% of absolute temperature

2.1.10 Vacuum Gage

The vacuum gage must be checked visually for damage and calibrated according to the directions in subsection 2.2.7. Any gage which is damaged and/or too erratic to be calibrated is rejected.

A vacuum gage with the following specifications is adequate for monitoring the sampling train vacuum:

Range:	0 to 76 cm Hg (0 to 30 in. Hg)
Divisions:	25 mm Hg (1 in. Hg)

2.1.11 Check Valve

A one-way check valve equipped with convenient-sized fittings is required in the sampling train. Visually check for stripped thread and blow alternately in each end to check for proper operation.

2.1.12 Valves

Two metering valves(1 ball and 1 needle) with convenient-sized fittings are required in the sampling train. Locate the valves in the sampling train and check for proper operation. Reject any valve that cannot be adjusted over the desired operating range.

2.1.13 Stack Gas Velocity Measuring System

See the Quality Assurance Document of this series for Determination of Stack Gas Velocity and Volumetric Flow Rate (type-S pitot tube) (ref. 3) for a discussion of this system.

2.1.14 Stack Gas Temperature Measuring System

This system is treated as a subsystem of the velocity measuring system and is discussed in the document referenced in the preceding subsection (i.e., 2.1.13). A more recent discussion is also found in reference 4. The temperature measuring system must be installed so that the sensor extends beyond the probe tip and does not touch metal. The position should be about 1.9 to 2.5 cm (0.75 to 1 in.) from the pitot tube and probe nozzle to avoid interference with the gas flow (fig. 2).

2.1.15 Stack Gas Pressure Measuring System

This system is treated as a subsystem of the velocity measuring system and is discussed in the document referenced in subsection 2.1.13.

2.1.16 Filter Media

The filter media are not reusable and all new filters must be visually checked for pinholes and tears. Any filter with a flaw is rejected. A Millipore AA, or equivalent, should be used. It is suggested that a Whatman 41 filter be placed immediately against the downstream side of the Millipore filter. In subsequent analysis, all filters must be analyzed.

2.1.17 Atomic Absorption Spectrophotometer

An atomic absorption spectrophotometer equipped with a N_2O/acetylene burner is required to measure the absorbance of beryllium at 234.8 nm (app. A). A Perkin-Elmer Model 303, or its equivalent, has been found acceptable to accomplish the analysis. A standard containing 3 μg/mℓ Be should give an absorbance reading of approximately 0.4 absorbance units.

2.2 EQUIPMENT CALIBRATION

2.2.1 Nozzle Tip Diameter

The inside diameter of each new nozzle should be measured with a micrometer to the nearest 0.025 mm (0.001 in.). Make three individual measurements using different diameters (rotate about 45° each time) and calculate an average diameter. The range (i.e., difference in smallest and largest values) of the 3 measurements should not exceed 0.1 mm (0.004 in.). Number the nozzle and record the number and diameter of the nozzle to the nearest 0.025 mm (0.001 in.) in the laboratory calibration logbook. Etch or permanently fix the average diameter on the nozzle. The nozzle should be checked visually for nicks, dents, corrosion, and out-of-roundness before each field test. Repair and recalibrate prior to use if any damage is detected.

2.2.2 Probe Heater and Filter Box Calibration

Set up the sampling train with the probe connected to the sampling box. With the nozzle detached and a flow rate of 0.021 m^3/min (0.75 ft^3/min), profile the temperature in the probe as the heat is increased. Determine the inlet and outlet temperatures of the probe at a determined minimum reference point corresponding to the stack gas temperature. If the inlet and outlet temperatures are not greater than the stack gas temperature, an adjustment must be made. If the probe is not equipped to continuously monitor its temperature, the temperature should be plotted as a function of percent of the power setting (ref. 5). It should be noted that outside skin temperature is only a pseudo temperature and will not be the actual temperature inside the probe.

A thermocouple with leads approximately 3 meters long can be used with a potentiometer readout to profile the probe temperature (draw the thermocouple along the inside length of the probe while maintaining a sample flow rate of 0.021 m^3/min. An alternate approach would be to use a remote reading thermometer.

The filter box temperature can be monitored with a thermocouple, thermistor, or dial-type thermometer. Adjust the voltage supply until the box temperature is greater than the minimum stack gas temperature. The temperature should not vary more than ±14°C (±25°F) over a 2-hour period. (Care should be taken as membrane filters deteriorate at temperatures greater than 107°C).

In all temperature calibrations, the reference points should be an ice bath and boiling water (corrected for pressure).

The probe and filter box heating units should be calibrated when new, after repairs, or any time the presampling check indicates that the system is not functioning properly.

2.2.3 Dry Gas Meter and Orifice Meter

An initial check should be made of the sampling train to check for proper operation of the pump, dry gas meter, vacuum gage, and dry gas meter thermometer(s). After the meter system components have been checked, the vacuum system should be leak-checked. This is done by plugging the inlet side of the metering system, pulling a vacuum of 635 mm Hg (25 in. of Hg), and observing the dry test meter. If the leakage exceeds .00057 m^3/min (0.02 ft^3/min), the leak(s) must be found and eliminated until the above specification is satisfied.

A second qualitative leak check is required if a diaphragm pump is used in the sampling train. One way to accomplish this check is to insert a valve into the train between the pump and the dry gas meter. With both the bypass valve and the inserted valve closed, pressurize the outlet side of the pump to the capacity of the pump. After the initial surge of bubbles in the impingers have subsided, all activity in the impingers should cease. If this phenomenon does not occur then further testing should be done to determine the source of the leak.

The dry gas meter should be calibrated when new, and a three-point check performed prior to each field trip. (The postcalibration check for one test serves as the precalibration check for the next test.) Calibration is performed by making simultaneous total volume measurements with a calibrated wet test meter and the dry gas meter in the meter box. The wet test meter must be of the proper capacity and accuracy. For commercial sampling trains, the wet test meter capacity must be at least 0.028 m^3/rev

($1\ ft^3$/rev) with an accuracy of ± 1 percent. The wet test meter must be of the proper capacity; otherwise, at the higher flow rates the linkage in the wet test meter will slip, producing an erratic correlation with the volume recorded by the dry gas meter. The recommended calibration procedures are as follows:

1. If a diaphragm pump and bypass valve are used, check the sampling system for valve float in the following manner (ref. 6). (The wet test meter is not required while checking for valve float in this manner.)

   a) Operate the sampling system at orifice readings between 1.3 mm and 130 mm H_2O (0.05 and 5 in. H_2O). Initially, operate the system at 0.0212 m^3/min (0.75 ft^3/min) for 10 minutes before taking data.

   b) Take data through the above range at flow rates of 0.002, 0.008, 0.014, 0.019, and 0.028 m^3/min (0.1, 0.3, 0.5, 0.7, and 1.0 ft^3/min), once with the bypass closed and once with it completely open. Time each setting for 1 minute. Record the ΔH setting (orifice reading) and the initial and final volumes on the dry test meter.

   c) Calculate ΔH@ from the two sets of data (see ref. 6).

$$\Delta H@ = \frac{K\ \Delta H}{V^2} \tag{1}$$

where $K = 4.48 \times 10^{-4}\ m^6$ in metric units, and $K = 0.56\ ft^6$ in English units. Plot two curves (one with the bypass valve opened and the other with it closed) of ΔH@ versus the volume (V) recorded by the dry test meter. If the valves are floating, the two curves will not coincide. Ideally, the curves should coincide and be horizontal over the entire range; in practice, the curve will probably have a slight slope.

This initial check for valve float is performed once after building or purchasing a sampling train and must be repeated whenever a new pump is installed in the system. Valve float, if present, must be corrected (ref. 6).

Date ________ Calibration By ________

Wet Test Meter No. ________

Barometric Pressure, P_b = ________ in.Hg

Wet Test Meter Capacity ________

Dry Gas Meter No. ________

Date of Wet Test Meter Calibration ________

Orifice manometer setting, ΔH, mm H_2O (in H_2O)	Gas vol. wet test meter V_w, $m^3(ft^3)$	Gas vol. dry gas meter V_d, $m^3(ft^3)$	Temperature				Time θ, min	Y	ΔH@
			Wet test Meter t_w, °C(°F)	Dry gas meter					
				Inlet t_{di}, °C(°F)	Outlet t_{do}, °C(°F)	Avg. t_d, °C(°F)			
2.5(0.1)	0.071(2.5)								
5.1(0.2)	0.071(2.5)								
7.6(0.3)	0.071(2.5)								
12.7(0.5)	0.14 (5)								
25.4(1.0)	0.14 (5)								
50.8(2.0)	0.28 (10)								
101.6(3.0)	0.28 (10)								
127.0(5.0)	0.28 (10)								
203.2(8.0)	0.28 (10)								
						Average			

Calculations

ΔH	$\frac{\Delta H}{13.6}$	Y $\frac{V_w P_b (t_d + 273)}{V_d \left(P_b + \frac{\Delta H}{13.6}\right) \left(t_w + 273\right)}$	ΔH@ $\frac{.0012\ \Delta H}{P_b\ (t_{do} + 273)} \left[\frac{(t_w + 273)\ \theta}{V_w}\right]^2$
0.1	0.00735		
0.2	0.0147		
0.3	0.0221		
0.5	0.0368		
1.0	0.0735		
2.0	0.147		
3.0	0.221		
4.0	0.294		
8.0	0.588		

Figure 3. Dry gas meter and orifice meter calibration and calculation form.

2. Determination of $\Delta H@$ and γ is as follows:

a) The wet test meter is placed upstream of the sampling system with its outlet connected to the inlet (sample umbilical connection) of the meter box (ref. 5). These connections must be leak-free.

b) Operate the pump for 15 minutes to warm up the pump and wet the surface of the wet test meter ($\approx 0.021\ m^3/min$).

c) Collect and record (as shown in figure 3) the calibration data by setting ΔH on the orifice manometer and letting a given volume of air pass through the wet test meter (the larger the volume, the greater the accuracy). Repeat the above procedures until the data are collected. Always have the bypass valve open. A stop watch or laboratory timer is used to record the elapsed time (θ) of the calibration.

The symbols in figure 3 are:

V_w = Gas volume passing through the wet test meter, m^3 (ft^3)

V_d = Gas volume passing through the dry test meter, m^3 (ft^3)

t_w = Temperature of the gas in the wet test meter, °C (°F)

t_{d_i} = Temperature of the inlet gas of the dry test meter, °C (°F)

t_{d_o} = Temperature of the outlet gas of the dry test meter, °C (°F)

t_d = Average temperature of the gas in the dry test meter, obtained by the average of t_{d_i} and t_{d_o}, °C (°F)

θ = Time of calibration run, minutes

ΔH = Orifice manometer setting, with a resultant orifice meter pressure drop, mm H_2O (in. H_2O)

γ = Ratio of volumetric measurement by wet test meter to dry test meter. Tolerance = ± 0.02

P_b = Barometric pressure, mm Hg (in. Hg)

$\Delta H@$ = Orifice meter pressure differential that gives a flow rate of 0.021 m^3/min (0.75 ft^3/min), mm H_2O (in. H_2O). Tolerance = ± 3.8 mm H_2O (0.15 in. H_2O)

d) Calculate γ and $\Delta H@$ for each orifice manometer setting and record on the calibration sheet as depicted in figure 3. The value of γ should be 1.0 ± 0.02; adjust the linkage of the dry test meter (if needed) as directed by the manufacturer until this tolerance is obtained. Plot curves of $\Delta H@$ versus ΔH (orifice manometer setting in mm of water). The value of $\Delta H@$ should be 46.7 ± 6.4 mm H_2O (1.84 ± 0.25 in. H_2O) with a variability no greater than ± 3.8 mm H_2O (0.15 in. H_2O) over the range of 13 to 203 mm H_2O (0.5 to 8 in. H_2O) of water across the orifice. If this is not obtained, adjust the orifice opening or replace the orifice as directed in reference 2 and recalibrate.

The value of $\Delta H@$ obtained at a flow rate of 0.021 m^3/min is etched on the orifice meter. The completed form in figure 3 is filed in the calibration log book.

2.2.4 Thermometers

Thermometers are calibrated against a mercury-in-glass thermometer with at least $1^{\circ}C$ divisions at two or three points as applicable. The points are an ice bath, room temperature, and boiling water (corrected for pressure). A thermometer should be calibrated when new and checked at one point before each field test. A calibration curve should be constructed if the test thermometer does not read within ± 1 percent of the mercury-in-glass thermometer (both readings in $^{\circ}K$).

Record all calibration data in the calibration logbook.

2.2.5 Stack Gas Velocity Measuring System

The procedure for calibrating a type-S pitot tube is given in the Quality Assurance Document of this series applicable to Method 2, Determination of Stack Gas Velocity and Volumetric Flow Rate (type-S Pitot Tube) (ref. 3), with one exception: the type-S pitot tube should be calibrated in the same configuration that it is to be used or the free space between the nozzle and pitot tube must be at least 1.9 cm (0.75 in.). If the sampling train is designed for sampling at higher than normal flow rates thereby requiring the use of larger size nozzles, the free space shall be set on the largest sized nozzle to be used.

The coefficient should not vary more than ± 5 percent of the average over the operating range. If the average coefficient is outside the range of 0.83 to 0.87 and a sampling nomograph is used in maintaining isokinetic conditions, corrections must be made as directed in subsection 2.4.3.1.

2.2.6 Stack Gas Temperature Measuring System

A temperature-measuring device attachable to a pitot tube and capable of measuring the stack gas temperature to within 1.5 percent of the minimum absolute stack gas temperature is required. A high-quality mercury bulb thermometer calibrated at ice water and boiling water (corrected for local pressure) temperatures and readable to the nearest .3°C (1°F) is an acceptable laboratory standard for calibration of temperature-measuring devices. The calibration procedure is contained in section 2.1.2.2 of the Quality Assurance Document of this series for Method 2, Determination of Stack Gas Velocity and Volumetric Flow Rate (type-S Pitot Tube) (ref. 3).

2.2.7 Barometer

The field barometer should be checked against a mercury barometer before each field test. If the two differ by more than ± 5.1 mm Hg (0.2 in. Hg) adjust, calibrate, or replace the field barometer as applicable. Record the results in the calibration logbook. Date and sign the entry.

2.2.8 Atomic Absorption Spectrophotometer

A spectrophotometer is required which is capable of measuring the absorption at 234.8 nm. A Perkin Elmer Model 303 or equivalent equipped with a N_2O acetylene burner is recommended.

2.2.9 Orsat Analyzer (optional)

A standard Orsat analyzer may be used at combustion sources for the determination of stack gas molecular weight. See the document of this series entitled "Gas Analysis for Carbon Dioxide, Excess Air and Dry Molecular Weight", based on Method 3 (ref. 7).

2.3 PRESAMPLING PREPARATION

2.3.1 Preliminary Site Visit (optional)

The main purpose of a preliminary site visit is to gather information to design and implement an efficient source test. Prior preparation will result in the prevention of unwarranted loss of time, expenses, and injury to test and/or plant personnel. A test plan conceived from a comprehensive set of parameters will result in more precise and accurate results. This preliminary investigation (on-site) is optional and not a requirement. An experienced test group can, in most cases, obtain sufficient information on the source through communications with the plant engineer. The information should include pictures (diagrams) of the facilities.

2.3.1.1 Process (Background data on process and controls). It is recommended that the tester become familiar with the operation of the plant before a preliminary site visit is made. Data from similar operations that have been tested should be reviewed if they are available and applicable.

2.3.1.2 Sampling Site Preparedness. The management of each facility tested should provide an individual who understands the plant process and has the authority to make decisions concerning plant operation to work with the test team. This would include decisions concerning whether the plant would be operated at normal conditions or at rated capacity. This individual or individuals will supervise installation of ports, sampling platform, and electrical power. If the above installations are already in existence, they should be examined for their suitability for obtaining a valid test and for overall safety conditions (ref. 8). If the sampling platform, port size, and locations are sufficient, the diameter, area of the stack, and wall thickness should be determined. If ports have to be installed, specify at least 8 cm (3 in.) ports with plugs. Ten centimeter (4 in.) ports are preferred. Port locations should be based upon Method 1 of the Federal Register (ref. 9). One electric drop should be available at the test facility with 120-volt, 20-ampere service.

2.3.1.3 Stack Gas Conditions. The following can be determined on the initial site survey, either by measurement or estimation:

1. $T_{s_{avg}}$ = Approximate stack gas temperature
2. P_s = Static pressure (positive or negative)
3. ΔP_{max} and ΔP_{min} = Maximum and minimum velocity pressure heads
4. B_{wo} = Approximate moisture content
5. M_s = Molecular weight calculated from approximate gas constituent concentrations.

The above parameters can be roughly determined using an inclined manometer with a range of 0-127 mm H_2O (0-5 in. H_2O), a type-S pitot tube, and a manual thermometer or thermocouple attached to the pitot tube with a potentiometric readout device. The moisture content (approximate) can be determined by the wet bulb-dry bulb method, and the gaseous constituents by hand-held indicator kits. Nomographs are useful in checking and estimating the preliminary required data (ref. 10).

2.3.1.4 Methods and Equipment for Transporting Apparatus to Test Site. Ropes, block and tackle, and other hoisting equipment should belong in the repertoire of any stack sampler. The initial site visit should include a preconceived plan between plant personnel and tester on how the equipment can best be transported to the sampling site. Electric forklifts should be utilized when at all possible. In addition to the above, it is recommended, when permissible, that pictures be taken of the hoisting and sampling areas, so that any further correspondence (either by letter or telephone) will be clarified.

2.3.2 Apparatus Check

Each item to be used should be visually checked for damage and/or excessive wear before each field test. Items should be repaired or replaced if judged to be unsuitable for use by the visual inspection.

Figure 4 shows a checklist for the three phases of a field test. It is meant to serve as an aid to the individuals concerned with procuring and checking the required equipment, and as a means for readily determining the equipment status at any point in time. The completed form should be dated, signed by the field crew supervisor, and filed in the operational logbook upon completion of a field test. This includes initiating the replacement of worn or damaged items of equipment. Procedures for performing the checks are given in the appropriate subsections of this operations manual; a check is placed in the proper row and column of the checklist as the check/operation is completed.

In addition to a visual check, the following performance and/or calibration checks are performed before each field test.

2.3.2.1 Sampling Train. The design specifications of the particulate sampling train used by EPA are described in APTD-0581 (ref. 2). Commercial models of this train are available. Each individual train must be examined to see if it is in compliance with the specifications in APTD-0581 (ref. 2) or its equivalent. In addition, the Office of Air Programs Publication No. APTD-0576 is a valuable source (ref. 5).

1. Nozzle. The nozzle is visually checked for damage, especially the sharp leading edge, and the tip opening is checked for out-of-roundness. If there is any sign of damage or out-of-roundness of the tip, the nozzle diameter should be calibrated according to subsection 2.2.1. Clean the nozzle by scrubbing with tap water, followed with a wash (2 hours) in 1:1 (V/V) hydrochloric acid-water wash and rinsed with distilled, deionized water. An alternate cleaning procedure when rust and/or organic materials

PRESAMPLING CHECKLIST

TEST SITE____________________ CREW SUPERVISOR______________________ DATE________________

Date and Initial Appropriate Block as Procedure is Completed

ITEM	Visual Check for Damage	Leak Check Sampling Train	Performance/ Calib. Check	Packed for Shipment
SAMPLING PREPARATION AND SAMPLING APPARATUS: 1. Nozzle				
2. Probe				
3. Filter Holder				
4. Filter Holder Box				
5. Filters				
6. Thermometers				
7. Impingers				
8. Vacuum Pump				
9. Dry Gas Meter				
10. Orifice Meter				
11. Inclined Manometer				
12. Vacuum Gage				
13. Check Valve				
14. Metering Valves				
15. By-Pass Valve				
16. Type-S Pitot Tube		▨		
17. Connecting Lines		▨		
18. Barometer		▨		
19. Inclined Manometer		▨		
20. Stack Temperature Measuring System		▨	▨	
SAMPLE RECOVERY: 21. Probe Brush		▨	▨	
22. Storage Containers		▨	▨	
23. Graduated Cylinders		▨	▨	
24. Wash Bottles		▨	▨	
REAGENTS: 25. Silica Gel		▨	▨	
26. Distilled Water		▨	▨	▨
27. Crushed Ice		▨	▨	▨
28. Filters		▨	▨	
DOCUMENTATION: 29. Data Sheets		▨		
CALCULATIONS & DATA VALIDATION: 30. Sampling Nomograph		▨		
31. Combustion Nomographs		▨		
32. Pocket Calculator		▨		
TOOLS AND EQUIPMENT: 33. Transportation Equipment		▨		
34. Safety Equipment		▨		
35. Tools and Spare Parts		▨		
36. Miscellaneous Supplies		▨		

Figure 4. Beryllium measurement checklist

ON-SITE MEASUREMENT CHECKLIST

TEST ______________ CREW SUPERVISOR ______________ DATE __________

Date and Initial Appropriate Block as Procedure is Completed

ITEM	Unpacked or Purchased On-Site	Assembled and Performance Checked	Dissasembled and Packaged for Shipment
SAMPLING PREPARATION AND SAMPLING APPARATUS: 1. Nozzle		LEAK CHECK SAMPLING TRAIN	
2. Probe			
3. Filter Holder			
4. Filter Holder Box			
5. Filters			
6. Thermometers			
7. Impingers			
8. Vacuum Pump			
9. Dry Gas Meter			
10. Orifice Meter			
11. Inclined Manometer			
12. Vacuum Gage			
13. Check Valve			
14. Metering Valves			
15. By-Pass Valve			
16. Type-S Pitot Tube			
17. Connecting Lines			
18. Barometer			
19. Inclined Manometer			
20. Stack Temperature Measuring System			
SAMPLE RECOVERY: 21. Probe Brush		▨	
22. Storage Containers		▨	
23. Graduated Cylinders		▨	
24. Wash Bottles		▨	
REAGENTS: 25. Silica Gel		▨	
26. Distilled Water		▨	▨
27. Crushed Ice		▨	▨
28. Filters		▨	
DOCUMENTATION: 29. Data Sheets		▨	
CALCULATIONS & DATA VALIDATION: 30. Sampling Nomograph		▨	
31. Combustion Nomographs		▨	
32. Pocket Calculator		▨	
TOOLS AND EQUIPMENT: 33. Transportation Equipment		▨	
34. Safety Equipment		▨	
35. Tools and Spare Parts		▨	
36. Miscellaneous Supplies		▨	

Figure 4. Beryllium measurement checklist (continued)

POST-SAMPLING CHECKLIST

TEST SITE____________________ CREW SUPERVISOR______________________ DATE___________

Date and Initial Appropriate Block as Procedure is Completed

ITEM	Inspect for Damage and/or Excess Wear	Accepted for Future Use	To Be Replaced
SAMPLING PREPARATION AND SAMPLING APPARATUS: 1. Nozzle			
2. Probe			
3. Filter Holder			
4. Filter Holder Box			
5. Filters			
6. Thermometers			
7. Impingers			
8. Vacuum Pump			
9. Dry Gas Meter			
10. Orifice Meter			
11. Inclined Manometer			
12. Vacuum Gage			
13. Check Valve			
14. Metering Valves			
15. By-Pass Valve			
16. Type-S Pitot Tube			
17. Connecting Lines			
18. Barometer			
19. Inclined Manometer			
20. Stack Temperature Measuring System			
SAMPLE RECOVERY: 21. Probe Brush			
22. Storage Containers			
23. Graduated Cylinders			
24. Wash Bottles			
REAGENTS: 25. Silica Gel	▨		
26. Distilled Water	▨		▨
27. Crushed Ice	▨	▨	▨
28. Filters	▨	▨	
DOCUMENTATION: 29. Data Sheets			
CALCULATIONS AND DATA VALIDATION: 30. Sampling Nomograph			
31. Combustion Nomographs			
32. Pocket Calculator			
TOOLS AND EQUIPMENT: 33. Transportation Equipment			
34. Safety Equipment			
35. Tools and Spare Parts			
36. Miscellaneous Supplies			

Figure 4. Beryllium measurement checklist (continued)

are present is to precede the tap water wash by soaking the nozzle in sulfuric acid. At all times protect the knife edge from being damaged. A damaged knife edge must be repaired or the nozzle discarded. Minor repairs may be done by the tester with a file and assorted tools. An alternate approach, especially with major damage, is to send the nozzle to a qualified machine shop.

2. Probe. Check the probe in the following manner:

a) Disassemble probe and check for breakage of inner liner or damage to other parts of probe.

b) Clean all metal parts with acetone.

c) Reassemble probe and clean inner liner with brush, using tap water and acetone. In extreme cases, the glass liner can be cleaned with a stronger cleansing agent. Following the initial wash, the glass liner (interior surface) should be soaked for 2 hours in a 1:1 (V/V) hydrochloric acid-water wash. The final rinse should be with distilled, deionized water and acetone (reagent grade) that has been analyzed for Be. The acid wash is easily accomplished by placing a glass female socket over the end of the male outlet of the probe. The acid is added to the inlet side, and the probe is allowed to stand vertically for the duration of the wash period.

d) Check to see if probe will heat to the desired temperature to prevent condensation. The probe temperature can be profiled with a remote reading thermometer or with a thermocouple with a readout device.

e) The probe should be sealed on the nozzle side and checked for leaks at a vacuum of 380 mm Hg (15 in. Hg).

f) Cover the open ends of the probe with serum caps or equivalent.

At temperatures greater than 260°C (500°F) or if asbestos string has been used as a gasket between the glass probe and the union holding the nozzle to the probe and probe sheath, the probability of leakage exists. Most stacks have a negative pressure; therefore, a leak would introduce

diluent air into the system and result in a low bias. This problem can be eliminated by:

a) Sealing the sheath from the outside air with a rubber stopper or its equivalent (ref. 2), and

b) Drilling a 0.3 mm (1/8 in.) hole in the sheath on the opposite side of the pitot tube just behind the nut.

This modification also prevents "out" gases resulting from deterioration of the probe from contaminating the stack sample (ref. 11).

3. Sampling Train Leak-Check. Assemble the sampling train as shown in figure 2. With all the impingers empty, leak-check the sampling train by plugging the probe inlet and pulling a vacuum of 380 mm Hg (15 in. Hg). **Leaks greater than 2 percent of the sampling rate (i.e., about 0.0006 m^3/min or 0.02 ft^3/min) as indicated by the dry gas meter should be found and corrected before continuing.**

Note 2: The leak-check cannot be made through the probe if asbestos string is used in the gasket. In this case, leak-check as described in 2.4.3.6. Following this initial leak-check at 280 mm (15 in.) Hg vacuum, connect the probe and leak-check at 25 mm (1.0 in.) Hg vacuum. A leakage rate greater than 0.00057 m^3/min (0.02 ft^3/min) is unacceptable.

Note 3: If using stopcock grease, use only the high temperature type.

2.3.2.2 Dry Gas Meter Calibration Check. After the sampling train leak-check has been satisfactorily completed in the system as directed, follow the same procedure as used in calibrating the dry gas meter (see subsec. 2.2.3); make runs at ΔH settings equivalent to flow rates of about 0.01, 0.02, and 0.03 m^3/min (0.50, 0.75, and 1.0 ft^3/min). Calculate γ for each run (see equation in fig. 3). If γ at either one of the three points falls outside the range of 1.0 ± 0.02, the dry gas meter should be (1) adjusted and recalibrated, (2) recalibrated and a calibration curve constructed, or (3) replaced. Record the results in the calibration logbook. Date and sign the entry.

2.3.2.3 Needle Valve(s) Check. The needle valve(s) should be disassembled and cleaned or replaced at any sign of erratic flow-rate behavior attributable to the needle valve as observed during the above checks or when unable to regulate the flow rate at desired levels. Document the adequacy of the needle valve with a check mark in the performance check column of the presampling checklist (figure 4).

2.3.2.4 Probe Heater Check. Connect the probe heating system. The probe should become uniformly hot to the touch within a few minutes after being turned on. If it does not heat properly, repair or replace as necessary. Document as part of the sampling probe performance check for the presampling phase (figure 4).

2.3.2.5 Filter Holder Box Heater. Check the heating system to verify that a temperature sufficient to prevent condensation can be maintained for at least 1 hour at laboratory conditions.

2.3.2.6 Barometer. The barometer is checked as a part of the stack gas pressure measuring system in the Quality Assurance Document of this series applicable to Method 2 (ref. 3).

2.3.2.7 Stack Gas Velocity Measuring System. Check the velocity measuring system according to the directions given in the Quality Assurance Document of this series for Method 2. Visual and performance checks are documented in figure 4 under visual check for damage and performance and/or calibration check for the presampling phase of the field test. If a calibration check is made, it should be recorded, dated, and signed in the calibration logbook.

2.3.2.8 Filter. The filter should be permanently numbered along its outside edge, where particulates (Be) will not be collected, for identification. Each lot of filters should be checked by the analytical technique for background concentration of Be. Results of the check are recorded in the laboratory logbook. The lot of filters should be rejected if the background Be is above the minimum detectable of the analytical technique. Filters are placed in inert containers and sealed with the filter number written on the container.

2.3.3 Package Equipment for Shipment

An important aspect of any source testing method in terms of logistics, time of sampling, and quality of data is the packing of equipment with regard to (1) accessibility in the field, (2) ease of movement on site, and (3) optimum functioning of measurement devices in the field. Equipment should be packed under the assumption that it will receive severe treatment during shipping and field operation.

2.3.3.1 Type-S Pitot Tube and Probe. Pack the pitot tube and probe in a case protected by expanded polyethylene or other suitable packing material. An ideal container is a wooden case or equivalent lined with expanded polyethylene in which separate compartments are cut to hold the individual devices. It is also recommended that inserts for the individual nozzles be provided. The case should have handles that can withstand hoisting and should be rigid enough to prevent bending or twisting of the devices during shipping and handling.

2.3.2.2 Differential Pressure Gage (Dual Inclined Manometer). Always close all valves on the pressure gage. Pack it in a suitable case for shipment. Spare parts, such as O-rings and gage oil (dual inclined manometer), should also be packed.

2.3.3.3 Stack Temperature Measuring Device. The temperature-measuring device (thermocouple, thermistor, remote reading thermometer, etc.) should be protected from breakage; i.e., placed in a tube or a suitable shipping container. If the device is an integral part of the pitot tube, it can be shipped in the type-S pitot tube shipping case.

2.3.3.4 Barometer. The barometer should be packed in a shock-mounted (spring system) carrying case.

2.3.3.5 Pitot Tube Lines and Sample Line (Umbilical). All pitot tube lines and sample lines should be coiled to utilize the smallest amount of space. The ends should be connected together and sealed to prevent dust and dirt from impairing their operation. For shipment all lines should be stored in a case (foot locker) for protection and portability.

2.3.3.6 Glassware (Impingers, U-Joints, etc.). A word of caution is needed in the use of glassware. It is expensive and fragile, but with sensible handling and packing its failure rate and resultant costs are minimal. Generally, breakage of glassware occurs during packing and movement to the sampling facility. It is recommended that glass impingers be packed in a suitable case with approximate dimensions of 50 x 50 x 50 cm (20 x 20 x 20 in.) with a three-tiered layer of expanded polyethylene in which holes are cut to hold the glass impingers. At least 13 mm (1/2 in.) of cushioning material should be placed in both top and bottom of the shipping case (ref. 12). A separate case lined with expanded polyethylene, with layers of 80 mm (3 in.) polyethylene, can be used to carry the rest of the individual glass joints, filter holders, and filters. One major point to consider in shipping cases is the construction materials. Durable containers, although more expensive to build, are the most cost effective in the long term. A poorly constructed shipping case of inferior material will quickly deteriorate.

2.3.3.7 Metering System (Meter Box Assembly). A standard (commercial unit including pump, vacuum gage, dry test meter, inclined manometer, etc., are contained in one meter box. This meter box should be placed in a shipping container lined with a cushioning material such as polyurethane.

If the vacuum pump is not integral to the meter box, it should be packed in a shipping container unless its housing is sufficient for travel. Additional pump oil should be packed with the pump if oil is required for its operation.

2.3.3.8 Sampling and Sample Recovery. Sampling and sample recovery equipment include the following:

1. Probe brush (commercial unit) or fabricated tube as long as the probe.
2. Glass wash bottles.
3. Leakless glass storage containers (500 mℓ) for collected samples and retention of blanks.
4. Graduated cylinder (250 mℓ).

5. **Silica gel: indicating type, 6-16 mesh, dried at 175°C (350°F) for 2 hours in preweighed lots of approximately 200 g (two/test).**
6. **Ice chest and water container.**
7. **Acetone: AA (atomic absorption) grade.**
8. **Distilled, deionized water.**
9. **Wash acid: 1:1 (V/V) hydrochloric acid-water solution.**

All glass bottles or glass storage containers should be packed with cushioning material at the top and bottom of the case with some form of divider to separate the components. One shipping case can contain the acetone, preweighed silica gel (plastic, for ≃ 300 mℓ), glass wash bottles, graduated cylinders, and probe brush. A water container and ice chest can be shipped as is. It is recommended in certain cases that these two items be purchased on-site. A general rule of thumb in source testing is "when possible, always carry a spare."

Prior to the field test all glass storage bottles and the graduated cylinders must be cleaned in 1:1 (V/V) hydrochloric acid water and rinsed with distilled, deionized water.

2.3.3.9 Source Sampling Tools and Equipment. The need for specific tools and equipment will vary from test to test. A listing of the most frequently used tools and equipment is given below:

1. Equipment Transportation

(a) A lightweight handtruck that can be used to transport cases and that can be converted to a four-wheel cart for supporting the meter box control unit.

(b) A 13 mm (1/2 in.) continuous filament nylon rope with large-throat snap hook and snatch block for raising and lowering equipment on stacks and roofs.

(c) Tarpaulin or plastic to protect equipment in case of rain. Sash cord, 6 mm (1/4 in.) diameter for securing equipment and tarpaulin.

(d) One canvas bucket, useful for transporting small items up and down the stack.

2. **Safety Equipment**

a) Safety harness with nylon and steel lanyards, large-throat snap hooks for use with lanyards for hooking over guard rails or safety line on stack.

b) A fail-safe climbing hook for use with climbing harness when climbing ladders having a safety cable.

c) Hard hats with chin straps and winter liners, gas masks, safety glasses and/or safety goggles, and first aid kit.

d) Protective clothing, including the following: appropriate suits for both heat and cold, gloves (both asbestos and cloth), and steel-toed shoes.

e) Steel cable, 5 mm (3/16 in.) diameter, with thimbles, cable clips, and turn-buckles. These are required for installing a safety line or for securing equipment to the stack structure.

3. Tools and Spare Parts

a) Electrical and Power Equipment

1) Circular saw
2) Variable voltage transformer
3) Variable speed electrical drill and bits
4) Ammeter-voltmeter-ohmeter (VOM)
5) Extension cords: Light (No. 14 AWG), 2 cords x 8 m (25 ft) long
6) 2-3 wire electrical adapters
7) 3-wire electrical triple taps
8) Thermocouple extension wire
9) Thermocouple plugs
10) Fuses
11) Electrical wire

b) Tools

1) Tool boxes (one large, one small)
2) Screwdrivers
   (a) One set flat blade
   (b) One set Phillips

        3) C-clamps (2): 150 mm (6 in.), 80 mm (3 in.)

    c) Wrenches

        1) Open end set: 6 to 25 mm (1/4 to 1 in.)
        2) Adjustables: 305 mm (12 in.), 150 mm (6 in.)
        3) One chain wrench
        4) One 305 mm (12 in.) pipe wrench
        5) One Allen wrench set

    d) Miscellaneous

        1) Silicone sealer
        2) Silicone vacuum grease (high temperature)
        3) Pump oil
        4) Manometers (gage oil)
        5) Antiseize compound
        6) Pipe fittings
        7) Dry cell batteries
        8) Flashlight
        9) Valves
        10) Dial thermometers, 150 mm (6 in.) and 915 mm (36 in.)
        11) Vacuum gage
        12) SS tubing: 6 mm (1/4 in.), 9 mm (3/8 in.), 13 mm (1/2 in.); short lengths
        13) Heavy-duty wire (telephone type)
        14) Adjustable packing gland

2.3.3.10 Data Recording. Pack one large briefcase with at least the following:

1. Nomograph for maintaining isokinetic conditions,
2. Data sheets or data notebook,
3. Carbon paper,
4. Slide rule or electronic calculator,
5. Psychrometric charts,
6. Combustion nomographs (ref. 10),

7. Pencils, pens, and
8. Calibration data, $\Delta H@$, γ, and C_p,

2.4 ON-SITE MEASUREMENTS

The on-site measurement activities include transporting the equipment to the test site, unpacking and assembling the equipment, confirming duct measurements and traverse points (such preliminary determinations should be accomplished in a site visit), molecular weight determinations of the stack gas, moisture content, setting of the nomograph, sampling, sampling recovery, and data recording. A sample data form is shown in figure 5. Every quantitative stack or measurement data should be recorded on such a form.

2.4.1 Transport of Equipment to the Sampling Site

The most efficient means of transporting or moving the equipment from floor level to the sampling site (as decided during the preliminary site visit) should be used to place the equipment on-site. Care should be exercised against damaging the test equipment during the moving phase. Utilization of plant personnel or equipment (winches and forklifts) in moving the sampling gear is highly recommended.

2.4.2 Preliminary Measurements and Setup

2.4.2.1 Duct Measurement. Measure the duct dimensions and determine the number of traverse points according to Method 104 (appendix A) or according to the latest revision of Method 1 which is contained in the Final Report for this contract.

2.4.2.2 Sample Box Logistics. Once the sampling points are selected and the probe has been marked with either a china marker or heat-bonding fiberglass tape (<370°C ≈ 700°F) the most efficient setup for the sample box must be determined. A poor choice will create a backbreaking and time-consuming sampling experience. Two rail systems exist for sampling, (monorails and duorails) in which the sampling box moves on a track(s). Each

TEST IDENTIFICATION

PLANT NAME ________________ TEAM SUPERVISOR ________________

LOCATION ________________ DATE OF TEST ________________

SOURCE ________________ TIME ________________

OPERATOR(S) ________________ RUN NO. ________________

SAMPLING SITE IDENTIFICATION

STACK NUMBER ________________

STACK DIMENSIONS:

d ________ m, L ________ m, W ________ m,

STACK AREA ____________ m^2

DISTANCE FROM UPSTREAM DISTURBANCE ________ m

DISTANCE FROM OUTLET OR DOWNSTREAM DISTURBANCE ________ m

APPARATUS IDENTIFICATION

NOZZLE DIAMETER ________ mm DIFFERENTIAL PRESSURE GAGE:

DRY GAS METER NUMBER ________ RANGE ________ mm H_2O

Y ________ DIVISION ________ mm H_2O

SAMPLING PROBE LENGTH ________ BAROMETER NUMBER ________

ORIFICE METER $\Delta H@$ ________ mm H_2O C_p ________

PITOT TUBE NUMBER ________

RUN INFORMATION

SAMPLE BOX NUMBER ____________ PROBE HEATER SETTING ________ °C

FILTER NUMBERS ____________ FILTER BOX HEATER SETTING ______ °C

SILICA GEL NUMBERS ____________

SAMPLING TRAIN LEAKAGE RATE ______ m^3/min at ________ mm Hg Vacuum

PRELIMINARY DATA FOR ISOKINETIC SAMPLING

ΔP_{avg} ________ mm H_2O B_{wo} ________

P_{bar} ________ mm Hg T_s ________ °K

P_g ________ mm Hg "C" FACTOR ________

$P_s = (P_{bar} + P_g)$ ________ mm Hg AMBIENT TEMPERATURE ________ °C

Figure 5. Sample data form for beryllium emissions determinations.

RECORDED TEST DATA

TRAVERSE POINT NUMBER	SAMPLING TIME θ(MIN)	STACK TEMP. (°C)	VELOCITY HEAD ΔP (mm H_2O)	$\sqrt{\Delta P}$	ORIFICE METER ΔH (mm H_2O)	DRY GAS METER READING (m^3)	GAS SAMPLE VOLUME V_m (m^3)	DRY GAS METER TEMPERATURE		FILTER BOX TEMP	GAS TEMP. OF CONDENSER OR IMPINGER (°C)
								INLET (°C)	OUTLET (°C)		
TOTAL			▨			▨				▨	▨
AVERAGE			▨			▨	▨			▨	▨

Figure 5. Sample data form for beryllium emissions determinations (continued).

VOLUME OF LIQUID COLLECTED (V_{1_c})

	Impinger Volume (mℓ)	Silica Gel Weight (g)	Total Volume Collected V_{1_c} (mℓ)
Final	mℓ	g	
Initial	mℓ	g	
Liquid Collected*	mℓ	g	
Conversion to mℓ**		mℓ	mℓ

* Liquid collected = Final - Initial

** Convert weight of water to volume of water by dividing total weight increase by density of water (1g/mℓ).

Figure 5. Sample data form for beryllium emissions determinations (continued).

individual sampling situation will dictate the system of the sample box support. It is recommended that the sampling box be modified to allow at least two alternate methods of support.

2.4.2.3 Stack Gas Moisture Content. Determine the approximate moisture content of the stack gases by Method 4 or its equivalent (ref. 13). If the particular source has been tested before or a good estimate of the moisture is available, this should be sufficient. The reference method uses the condensate collected during the sampling for the moisture content used in final calculations.

2.4.2.4 Molecular Weight of Stack Gas. Determine the dry molecular weight of the gas stream by Method 3 (ref. 7). It is recommended that the sample be of the integrated type for two reasons: 1) the possibility of a more representative sample and, 2) the convenience of taking the sample at the stack and being able to transport the sampling bag to a more suitable area for Orsat analysis.

2.4.2.5 Stack Temperature and Velocity Heads. Set up and level the dual inclined manometer and determine the minimum and maximum velocity head (ΔP) and the stack temperature (T_s). This is done most efficiently with a type-S pitot tube, with a temperature-sensing device attached. The ΔP's are determined with an inclined manometer by drawing the pitot tube across the stack diameter in two directions (circular stack with 90 ° traverses). This must be done in order to pick the correct nozzle size and to set the nomograph. Incorrect selection of nozzle size and/or setting of the nomograph may result in not being able to reach the isokinetic rate, thereby voiding the sample. Determine the static pressure as directed in the Quality Assurance Document of this series for Method 2 (ref 3). If the stack temperature is greater than 93°C (200°F), arrangements must be made to prevent deterioration of the filter, e.g., move the filter to a position between the third and fourth impingers.

2.4.3 Sampling

The on-site sampling includes making a final selection of proper nozzle size, setting the nomograph (if used), loading the filter into the filter holder, preparing and assembling the sampling train, making an initial leak-check, inserting the probe into the stack, sealing the port, sampling isokinetically while traversing, recording the data, and making a final leak-check of the sampling system. Sampling is the foundation of source testing. Critical problems in testing result from poor or incorrect sampling more frequently than from any other part of the measurement process. The analytical process (laboratory) can never correct for errors made in the field resulting from poor judgment or instrumental failure. If the initial site survey, apparatus check and calibration, and preliminary measurement and setup on-site have been implemented properly, the testing should go smoothly with a minimal amount of effort and crises.

2.4.3.1 Preliminary Setting of the Nomograph. The setup of the nomograph using the parameters obtained in subsection 2.4.2 is given in detail in appendix B of this document. A procedure is included in the appendix for checking the nomograph for correct design (accuracy).

> Note 4: If the coefficient, C_p, of the type-S pitot tube being used is outside the range of 0.85 ± 0.02, compute the ratio $(C_p/0.85)^2$ and multiply this constant times the correction factor, C, obtained from the nomograph. Use this new "C" factor in setting the nomograph for isokinetic sampling (see appendix B for further discussion).

2.4.3.2 Selection of Nozzle Size. After the nozzle size and appropriate probe length have been selected, insert the nozzle in the probe sheath union and tighten the union. Do not use wrenches; finger-tight is sufficient in most cases. Using uncontrolled pressure in tightening the union will result in a broken or cracked inner-liner. Keep the ball joint and nozzle tip protected from dust and dirt with a serum cap or equivalent.

2.4.3.3 Assembling of Sampling Train. Assemble the glass impinger train as follows:

1. Measure 200 mℓ of distilled water in the graduated cylinder 5-mℓ divisions) and place approximately 100 mℓ of the water in each of the first two impingers
2. The third impinger is left empty.
3. Place approximately 200 g of preweighed indicating silica gel into the fourth impinger.

The first, third, and fourth impingers are modified Greenburg-Smith while the second impinger is of the standard Greenburg-Smith design. Place the impingers into the sample box and assemble the sampling train using the appropriate U-joints. A very light coat of silicone grease (acetone insoluble) should be applied carefully to the joints, to avoid leaks in the system. Depending upon the design of the impinger, apply the lubricant in a manner that prevents contact with the sample.

The loading of the impingers into the sample box can be done in the laboratory by sealing the inlet to the first impinger, the outlet of the third impinger, and inlet and outlet of the silica gel impinger. This is practical only when the sampling site is near and the logistics are suitable.

The impingers must be maintained in an ice bath during sampling to remove the condensibles and to keep the exit gas at or below 20°C (68°F).

2.4.3.4 Loading of Filter. Load the filters as follows:

1. The filters are removed from their sealed container and placed in the filter holder with the Millipore filter toward the probe. The filter should have an identification number, and the filter holder should be numbered with a semipermanent marker to preserve the integrity of the sample Make certain that the filters are centered correctly in the holder. The filter should be tightened until the two halves are secure. Overtightening the two halves can break the filter holder or tear the filter.

2. Place the filter holder into the sample box and connect the exit of the filter to the inlet of the first impinger. Plug the inlet of the

filter with a glass ball to check for leaks. Connect the meter box (vacuum port) to the sample box with the appropriate umbilical cord.

2.4.3.5 Leak-Check. Leak-check the sampling train by plugging the inlet to the filter holder, turning on the vacuum pump, and opening the valve system until the vacuum in the system reaches 381 mm Hg (15 in. Hg). A leakage rate not in excess of 0.0006 m^3/min (0.02 ft^3/min) at 381 mm Hg is acceptable. Release the pressure in the system but do not turn off the pump until the following sequence has been completed:

1. Slowly release the pressure in the system by carefully opening (twisting) the glass ball in the inlet of the filter holder.
2. Shut the coarse valve (main vacuum valve).
3. When the vacuum gage reads zero vacuum, remove the glass ball and shut down the pump.

2.4.3.6 Installation of Probe. Mount the probe in the sampling box and connect the probe to the inlet of the filter and leak-check in the following manner:

1. Seal the inlet of the probe nozzle with a serum cap.
2. Turn on vacuum pump.
3. Open the valve system and adjust the vacuum to 381 mm Hg (15 in. Hg).
4. Check the leakage rate on the dry gas meter. A leakage rate not in excess of 0.0006 m^3/min (0.02 ft^3/min) at 381 mm Hg (15 in. Hg) is acceptable.

   Note 5: If an asbestos string is used in the fabrication of the probe nozzle to the probe liner connection, leak-check at 25 mm Hg (1 in. Hg) vacuum only. If a leak-free connection in the nozzle is employed, the total train, filter and probe, can be initially checked at 381 mm Hg (15 in. Hg) vacuum.

5. After completion of the leak check, release the pressure as follows:

   a) Slowly release vacuum by carefully opening (squeezing) the serum cap until the system pressure is back to ambient (monitor with built-in vacuum gage).

b) Turn valve system off (coarse valve).

c) Turn off the vacuum pump.

Operations in subsections 2.4.3.5 and 2.4.3.6 can be combined, thereby requiring only one initial leak-check. Record leakage rate on the form in figure 3.

2.4.3.7 Taking of Sample. Turn on the sample box and fill the impinger train container with crushed ice. The meter box operator should now recheck the setting of the nomograph while the sample box operator checks the filter box temperature gage to confirm that it is coming up to operating temperature; likewise, he can touch the probe to see if it is heating. It is recommended that a thermocouple be mounted next to the glass liner so that the probe temperature can be monitored.

As soon as the filter box temperature (if applicable) and the probe temperature have reached the minimum stack gas temperature, commence sampling.

1. Remove the plug or cap from the sampling port and remove the dust (particulates) on the port walls by utilizing a wire brush or its equivalent. Remove the serum cap from the nozzle tip. Record the initial volume of the test meter on the data log sheet of figure 5.
2. If the sample gas is hot, start at the traverse point farthest from the port and draw the probe out as the test continues. Asbestos gloves should be used in handling hot sampling probes and pitot tubes.

   Note 6: If the stack gas temperature is $\geq$ 93°C (200°F) in all probability the filter holder should be placed between the third and fourth impingers.
3. Attach a proper electrical ground to the probe and sampling system.
4. Insert the probe to the farthest traverse point with the nozzle pointing directly into the gas stream. Seal the port and immediately start the pump. Adjust the coarse and fine control valves

until isokinetic conditions are obtained. Note the time and record it on the data log sheet of figure 5.

5. Maintain isokinetic conditions during the entire sampling period. Sample for an equal amount of time at each traverse point. The time period at each traverse point should be long enough to set the sample rate and record the required data. The time period at each traverse point must be long enough to obtain a total sampling period representative of the process being monitored. While sampling, reset the nomograph if:

a) The temperature in the stack changes more than ± 14°C (25°F) for $T_s < 793°C$ (1460°F) or ± 28°C (50°F) for $T_s > 793°C$, or

b) T_m (average temperature of meter) varies more than ± 6°C (11°F).

Adjust the sampling rate for every point and maintain the isokinetic rate by continuous observation. Record the meter volume when sampling has been completed at each individual traverse point. Take readings at each sampling point, at least every 5 minutes (or during sampling period at each traverse point): all the readings and adjustments should not be attempted for time intervals of less than 2 minutes. When significant changes in stack conditions are observed, compensating adjustments in flow rate should be made to maintain isokinetic conditions. Record on the data log sheet of figure 5 the traverse point number, stack temperature (T_s), velocity pressure head (ΔP, mm H_2O or in. H_2O), (orifice pressure differential (ΔH, mm Hg or in. H_2O), gas temperature at dry gas meter ($T_{m_{in}}$) and $T_{m_{out}}$ or T_{avg}, °C or °F), sample box temperature, condenser temperature, and the probe temperature if the probe has a thermocouple and appropriate readout.

6. When sampling at one traverse point has been completed, move the sampler to the next point as quickly as possible. Close the control valve only when transferring the sampler from one sample port to the other. Exclude the time required to transfer the sampler from one port to another from the total sampling time.

Upon transfer of the sampler to another port, the following procedures should be followed:

a) Monitor the vacuum through the system. An increase of vacuum is an indication of particulate buildup on the filter, collapse of the membrane filter, or plugging of the last impinger by wet silica gel. Loss in vacuum is an indication of a broken impinger, connector, filter, or a loose connection.

b) Keep the impingers iced down (i.e., monitor the condenser temperature) to hold the temperature below 20°C (68°F). Add salt to the ice bath if necessary.

c) Check the line voltage with a voltmeter if a digital temperature system is utilized.

> Note 7: Digital temperature systems may read erroneously with a drop in line voltage and/or interference from electromagnetic fields.

d) Make sure that the dual inclined manometer is level and that the pitot tube and pitot tube lines are unobstructed. A signal of trouble would be ΔP's that are not representative of the velocity heads obtained in a velocity traverse made during a preliminary site visit.

e) All data should be recorded on a data log sheet as depicted in figure 5.

7. At the completion of the test, close the coarse control valve on the meter, remove the probe from the stack, and turn off the pump. Remove the probe carefully from the stack, making certain that the nozzle does not scrape dust from the inside of the port. Keep the nozzle elevated to prevent sample loss. Place a serum cap or equivalent over the nozzle tip and leak-check the system at 51 mm Hg (2 in. Hg) vacuum above the operating vacuum during the test. The vacuum during the leak check should be no greater than 381 mm Hg (15 in. Hg). (Do not boil the water in the impingers.) Follow the same leak-check procedures as outlined in subsection 2.4.3.5. Seal the end of the nozzle. Disconnect the

pitot tube lines and umbilical. Protect the pitot tube and umbilical connections with tape or an appropriate equivalent. Record on the data log sheet of figure 5 the leakage rate in m^3/min (ft^3/min) and the vacuum at which the leak check was performed. Check all connectors such as umbilical connection, pitot tube lines, glass connections, etc. for evidence of malfunction. Record all abnormalities on the data log sheet. The logging of abnormalities will not necessarily void the sample, but it may help to improve the quality of sampling performance.

2.4.4 Sample Recovery

The reference method requires a quantitative transfer of the impinger solutions and filters to suitable storage containers. These transfers should be performed in a "laboratory-type" area to prevent contamination of the test samples. Move the sampling train and probe to the sample recovery area. Care should be taken to prevent loss or contamination of the samples. If the probe must be removed before moving to the recovery area, the probe should be sealed at both ends (serum caps), and the inlet of the filter plugged with a glass ball.

2.4.4.1 Container No. 1. Wipe the exterior of the filter holder surface to remove any excess dust or extraneous material. Remove the filter from the holder, place the filters in a precleaned glass container, and seal it. It is recommended that a piece of paper (smooth surface) be placed under the filter holder as the filter is being removed to prevent loss of sample. Removal of the filter is more efficient if tweezers are utilized. Teflon-tipped tweezers and a Teflon or a clean stainless steel scalpel should be used to handle filters. If a filter is torn, all pieces must be saved. Any loose particulate matter (or filter material clinging to the frit) should be placed in this container. Record date, time of test, location of test, and the number of the run on this container. This data should also be recorded on the data log sheet of figure 5.

2.4.4.2 Container No. 2. Wash all internal surfaces of the filter with distilled, deionized water and atomic absorption grade acetone. Determine the volume of liquid (acetone and water) and place in the container.

Note 8: An alternate procedure for determining the amount of liquid (acetone and water) is by weighing (± .5g). A balance accurate to ± 1 g is sufficient.

It is recommended that the probe be washed by attaching a graduated cyclone flask to the end of the probe and washing the probe contents into the container. This wash may require emptying the flask into container No. 2 several times. A brush (length ≥ probe length) should be used to loosen particulate matter. Record total volume of acetone wash, date, time of test, location, and run number on the container and on the data sheet of figure 5. Measure the total volume in the first three impingers by transferring their contents into a graduate with a measurement accuracy of ± .6 percent. This data should be recorded on container No. 2 and on the data log sheet of figure 5. Rinse the first three impingers with water and acetone and transfer to the graduate. Transfer the solution in the graduate into container No. 2. All measurements are read to the nearest mℓ for correction for blanks in the final analysis. Place at least 300 mℓ of acetone and 300 mℓ of the water into separate properly identified sample containers for blanks. Record total volume of water, acetone wash, date, time of test, location, and run number on the sample and blank containers and on the data sheet of figure 5.

2.4.4.3 Container No. 3. Transfer the silica gel from the fourth impinger into its original preweighed container. Label the container with date, time of test, location, and any other pertinent data. This information should also be recorded on the data log sheet. All sample containers should be glass with caps lined with Teflon.

2.4.5 Sample Logistics (Data) and Packing of Equipment

The above procedures are followed until the required number of tests are completed. The following is recommended at the completion of testing.

1. Check all sample containers for proper labeling (time and date of test, location of testing, number of test, and any other pertinent documentation). This function should be performed at the end of each individual test or prior to such test if the impingers are to be utilized in further tests before returning to the laboratory.

2. All data recorded during field testing should be recorded in duplicate (carbon paper). One set of data should be mailed to the base laboratory, the other hand-carried. This precaution can prevent a very costly mistake.

3. All sample containers should be properly packed in a sample box for shipment to the base laboratory. All boxes should be properly labeled to prevent loss of the samples.

2.4.6 Data Validation

Following the directions given in subsection 2.5.3, calculate and/or determine the following:

1. Moisture content of the stack gas, B_{wo}.
2. Stack gas molecular weight on a wet basis, M_s.
3. The average stack gas velocity, $(v_s)_{avg}$.
4. The percent of isokinetic sampling for the sample run, I.

Compare these measured values to theoretical values derived from combustion nomographs (ref. 10) or to values obtained by other measurement methods, e.g., measuring B_{wo} by the wet bulb-dry bulb method.

Any large inexplicable differences in measured and theoretical values should be noted and special care taken to reduce the variability of that specific parameter for the next run. If the percent of isokinetic sampling is outside the range of 0.90 to 1.10, the run should be repeated. If it is known that the particle size distribution is below about 5 μm, the EPA Administrator may option to accept the data even if I is outside the interval of 100 ± 10 percent.

Values of B_{wo} and M_s as measured by the first sample run should be used in setting isokinetic conditions for subsequent runs unless there is reason to doubt their validity when compared with the values derived from preliminary measurements or estimates.

2.5 POSTSAMPLING OPERATIONS (Base Laboratory)

2.5.1 Apparatus Check

2.5.1.1 Type-S Pitot Tube. The type-S pitot tube is checked according to the Quality Assurance Document of this series for Method 2.

2.5.1.2 Dry Gas Meter and Orifice Meter (Sampling Train). A postcheck (a postcheck for one test can serve as the presampling check for the next test) should be made of the sampling train to check for proper operation of the pump, dry test meter, vacuum gage, and dry test meter thermometers. Leak-check the vacuum system. Determine γ and $\Delta H@$ at three points in the operating range. This not only checks on the system for future testing but also gives confidence in the data from the previous field test. This is a recommended procedure to improve the data quality and to prevent field sampling under assumed conditions.

2.5.2 Analysis (Laboratory)

The requirement for a precise and accurate analysis requires an experienced analyst and familiarity with the analytical method; calibration is of the utmost importance, and neglect in this area is unacceptable. The analytical method is based upon an acid digestion (perchloric acid, etc.), followed by the determination of Be at the 234.8-nm line utilizing an atomic absorption spectrophotometer.

2.5.2.1 Apparatus

1. Atomic Absorption Spectrophotometer. An instrument as described in subsection 2.1.17.
2. Hotplate. A general duty hotplate is required for evaporation and digestion of samples.
3. Perchloric Acid Fume Hood. The laboratory hood or fume cupboards should be noncombustible, constructed of metal or stoneware, and left either unpainted or protected with an inorganic coating such as porcelain. Glycerinelitharge should not be used as a sealant

on hood end blower systems; tetrafluoroethylene plastic and fluorocarbon greases are the best materials for this service. Only a fluorocarbon grease should be used as blower lubricant. Any other type of lubricant should be considered hazardous (ref. 8).

4. Pipettes. An ample number of (1, 3, 5, 8, 10 mℓ) pipettes (volumetric, Class A) are required for preparing standards and samples for analysis.
5. Volumetric Flasks. All volumetric glassware for preparing standards and samples should be volumetric, Class A.
6. Analytical Balance. An analytical balance that weighs to 0.1 mg is required to prepare standards.
7. Aspirating Bulb. Bulbs should be utilized in pipetting all solutions.

2.5.2.2 Reagents (Calibration and Analysis)

1. Water: deionized, distilled
2. Acetone: atomic absorption (AA) grade
3. Hydrocloric Acid: concentrated (ACS grade)
4. Nitric Acid: concentrated (ACS grade)
5. Perchloric Acid: concentrated, 70 percent (ACS grade)
6. Beryllium Powder: 98 percent minimum purity

Note 9: In lieu of beryllium powder, beryllium salt ($Be(NO_3)_2 \cdot 3H_2O$) of 98 percent purity can be utilized.

2.5.2.3 Standard Beryllium Solution (Stock Solution)

1. Prepare a 12 *N* sulfuric acid solution by adding 33.3 mℓ of concentrated H_2SO_4 to 66.6 mℓ H_2O in a 100 mℓ volumetric flask.
2. Dissolve 100.0 mg of the beryllium metal, weighed on an analytical balance, in 80 mℓ of the 12 *N* sulfuric acid solution and dilute to a volume in a 1000-mℓ volumetric flask (class A) with distilled, deionized water to give a 100-μg/mℓ Be standard.
3. Prepare a typical working analytical curve by pipetting 1, 2, 3, and 4 mℓ of the stock solution into four 100 mℓ volumetric flasks

and diluting to volume with 25 percent V/V hydrochloric acid, giving concentrations of 1, 2, 3, and 4 μg/mℓ, respectively. These dilute standards should be prepared fresh daily.

Note 10: If interferences such as aluminum (500 μg/mℓ) or high concentrations of silicon and magnesium are present, the standards prepared must contain the same reagents and major matrix elements as present in the sample solutions. Oxine (8-hydroxy quinoline) has been added to samples and standards to control interferences (ref. 14).

2.5.2.4 Working Analytical Curve. General guidelines for preparing a working analytical curve are:

1. Prepare standard solutions from the 100-μg/mℓ solution in suitable class A volumetric flasks.
2. Set up the atomic absorption instrument as directed by the manufacturer at the 234.8-nm line for determination of Be utilizing a $N_2O \cdot C_2H_2$ flame.
3. Establish the working range of the instrument (linearity). A typical linear calibration curve is obtained from 0 to 4 μg/mℓ. A standard containing 3 μg/mℓ Be will give an absorbance reading of approximately 0.4 absorbance units.

Note 11: If the concentrations of the field samples are below 1 μg/mℓ, a calibration curve shall be prepared covering the range from 0 to 1 μg/mℓ. In most cases this will require scale expansion.

2.5.2.5 Sample Preparation. The sample preparation is a wet digestion in concentrated perchloric acid.

1. Container No. 1. Transfer the filters and any loose particulate matter from the container into a 150-mℓ beaker. Add 35 mℓ concentrated nitric acid to the sample container, then transfer to the 150-mℓ beaker. Heat on a hotplate until light-brown fumes are no longer present to destroy all organic matter.

Note 12: The analyst must insure that the sample is heated until light brown fumes are no longer given off after the initial nitric acid addition; otherwise, dangerous organic perchlorates may result from the subsequent perchloric acid digestion. All organic matter must be destroyed before the addition of perchloric acid. In some cases the nitric acid addition and heating until brown fumes are no longer given off will have to be repeated to remove organic residues.

Note 13: Perchloric acid should be used only under a perchloric acid hood. The existing ductwork must be washable to prevent the buildup of dangerous perchlorates.

Note 14: The presence of a black residue in the beaker indicates incomplete oxidation. If an organic residue remains, add 50 mℓ nitric acid and repeat digestion.

Cool to room temperature and add 5 mℓ concentrated sulfuric acid and 5 mℓ concentrated perchloric acid; then follow instructions in subsection 2.5.2.6.

2. Container No. 2. Place a portion (50-75 mℓ) from Container No. 2 into a 150-mℓ beaker and put on a hotplate. Add a couple of glass boiling beads to minimize bumping. Add portions of the remaining solution as evaporation proceeds and evaporate to dryness. Do not leave the beaker on the hotplate after the liquids have evaporated. Cool the residue and add 35 mℓ of concentrated nitric acid. Heat the acid solution on a hotplate as directed in (1) above. Cool to room temperature and add 5 mℓ concentrated sulfuric acid and 5 mℓ perchloric acid. Then continue to subsection 2.5.2.6.

2.5.2.6 Sample Analysis. Samples from subsection 2.5.2.5 may be combined here for ease of analysis. A complete transfer when combining samples should be done with a 1:1 (V/V) sulfuric acid-perchloric acid rinse solution.

1. After transfer, place on a hotplate and evaporate to dryness in a perchloric acid hood.
2. Cool the residue.
3. Dissolve the residue in 10 mℓ of 25 percent V/V hydrochloric

acid. The sample is now ready for analysis.

Note 15: If prior experience suggests a concentrated solution exceeding the linear range of the working analytical curve, do not combine the two sample solutions (2.5.2.5). An alternative to this approach is a further dilution of the concentrated sample with 25 percent V/V hydrochloric acid. When employing this latter procedure, aspirate a minimum volume of the original sample solution before dilution. The dilution would require an aliquot from the original sample diluted to volume with 25 percent V/V hydrochloric acid. The dilution ratio is dependent upon the sample concentrations and must be determined by the analyst.

4. Analyze the samples at 234.8 nm using a nitrous oxide-acetylene flame. When utilizing a nitrous oxide-acetylene flame, allow the burner to warm up without aspiration for several minutes. This will help to reduce carbon formation. After continuous operation of the nitrous oxide burner head, deposits will build up near the slot causing a ragged flame. The burner slot should be cleaned at frequent intervals or whenever the flame becomes ragged. Report results in μg/mℓ and record the total volume of 25 V/V hydrochloric acid to the nearest mℓ. In recording the volume of solution, any dilution factors must be incorporated for subsequent calculations to determine the total μg of Be in the original sample volume.

Note 16: Aluminum, silicon, and other elements interfere with this method if present in large quantities. Standard methods are available to effectively eliminate these interferences (refs. 14, 15). Three ways are most commonly utilized:

(a) Standards matching the sample composition may be prepared. Utilization of this method suggests that the analyst has some independent knowledge of the sample history. This method should not be used unless the overall composition of the sample is known and its composition does not vary from sample to sample.

b) Addition of buffer solutions to both standards and samples.

c) The use of additive methods.

In most analyses a combination of methods two and three will yield the most precise and accurate analysis. Several things must be taken into consideration when an additional method (c) is utilized:

(1) The absorbance must be linear over the concentration range being analyzed.

(2) Chemical interferences must be constant over the range of metal concentrations.

(3) It is assumed that the entire absorption signal is due to the element being absorbed. This assumption must be verified for a sample series by checking the absorption at a nearby non-absorbing line (i.e., for the element being analyzed for) or by utilizing a background correction with a continuous source.

5. Blank preparation and analysis. A 300-mℓ water blank (distilled, deionized water used in the field) and a 300-mℓ acetone blank (acetone used in field sampling) should be prepared and analyzed as described in subsections 2.5.2.5(2) and 2.5.2.6(2,3). Report results in μg/mℓ Be for both water and acetone.

Standards should be interspersed with samples and blanks since the calibration can change slightly with time. The standard working curves should be run prior to and after sample analysis.

In considerations of the fact that the major source of beryllium is the mineral beryl ($3\ BeO \cdot Al_2O_3 \cdot 6SiO_2$) which includes two of the major interferences (i.e., Al and Si) in the atomic absorption method the following is recommended:

a) Field samples should be analyzed by the standard procedure as outlined in this document.

b) In addition to the above, a concentrated standard solution of Be should be added to the sample in microliter portions to check for interferences by method of standard additions (ref. 14).

If the analyzed values for Be by the standard procedures and the method of standard additions differs by 25 percent (after background correction), it is suggested that 8 hydroxyquinoline be added to samples and standards (ref. 13) to control interferences and the method of standard additions be employed as discussed in note 12 above.

2.5.2.7 Moisture Content (Silica Gel). Weigh the original preweighed container No. 3 containing silica gel utilized in the field. Record the final weight in a table such as figure 5.

2.5.3 Calculations

Calculation errors due to procedural or mathematical mistakes can be a large component of total system error. Therefore, it is recommended that each set of calculations be repeated or spot-checked (every third calculation), preferably by a team member other than the one who performed the original calculations. If a difference greater than a typical round-off error is detected, the calculations should be checked step by step until the source of error is found and corrected. A computer program is advantageous in reducing calculation errors. A standardized computer program could be developed to treat all raw field data. If a computer program is used, the original data entry should be checked, and if differences are observed, a new computer run made. Figure 6 shows a sample form which can be used to log computations.

2.5.3.1 Volume of Water Vapor, V_{w_s}. The volume of water vapor in the gas sample at stack conditions is calculated using the V_{1_c} value determined from the table in figure 5 and the following equation:

$$V_{w_s} = K_w V_{1_c} \frac{T_s}{P_s} \qquad (2)$$

where V_{w_s} = Volume of water vapor in the gas sample at stack conditions m^3 (ft^3)

CALCULATIONS AND DATA ANALYSIS

(1) VOLUME OF WATER VAPOR (Stack Conditions)

$$V_{w_s} = .00346\ V_{1_c} \frac{T_s}{P_s} = \underline{\qquad\qquad}\ m^3$$

where V_{1_c} = ______ mℓ P_s = ________ mm Hg

T_s = ______ °K

(2) DRY GAS VOLUME (Stack Conditions):

$$V_{m_s} = V_m \frac{T_s}{T_m} \frac{\left(P_{bar} + \frac{\Delta H_{avg}}{13.6}\right)}{P_s} = \underline{\qquad\qquad}\ m^3$$

where V_m = __________ m^3 P_{bar} = __________ mm Hg

T_s = __________ °K ΔH_{avg} = __________ mm H_2O

T_m = __________ °K P_s = __________ mm Hg

(3) DRY GAS VOLUME (Standard Conditions):

$$V_{m_{std}} = .3874\ V_m \frac{\left(P_{bar} + \frac{\Delta H_{avg}}{13.6}\right)}{T_m} = \underline{\qquad\qquad}\ m^3$$

where V_m = ________ m^3 P_{bar} = ________ mm Hg

T_m = ________ °K ΔH_{avg} = ________ mm H_2O

(4) TOTAL GAS VOLUME (Stack Conditions):

$$V_{total} = V_{m_s} + V_{w_s} = \underline{\qquad\qquad}\ m^3$$

where V_{m_s} = __________ m^3

V_{w_s} = __________ m^3

(5) MOISTURE CONTENT OF STACK GAS:

$$B_{wo} = \frac{0.0013\ V_{1_c}}{\left(V_{m_{std}} + 0.0013\right)} = \underline{\qquad\qquad}\ \text{(dimensionless)}$$

where V_{1_c} = __________ mℓ

$V_{m_{std}}$ = __________ m^3

Figure 6. Sample calculation and data analysis form

(6) MOLECULAR WEIGHT (Wet Basis):

$$M_s = M_d\ (1-B_{wo}) + 18\ B_{wo} =$$ ____________ g/g-mole

where

$M_d =$ ____________ g/gm-mole

$B_{wo} =$ ____________ g/g-mole

(7) AVERAGE STACK GAS VELOCITY:

$$(v_s)_{avg} = 34.96\ C_p\ (\sqrt{\Delta p})_{avg} \sqrt{\frac{(T_s)_{avg}}{P_s M_s}} =$$ ________ m/sec

where

$C_p =$ ____________ (dimensionless)

$P_s =$ ____________ mm Hg

$(\sqrt{\Delta p})_{avg} =$ ____________ mm $H_2O^{1/2}$

$M_s =$ ________ g/g-mole

$(T_s)_{avg} =$ ____________ °K

(8) BERYLLIUM COLLECTED:

$$W_t = V_\ell C_\ell - V_w C_w - V_a C_a =$$ ____________ μg

where

$V_1 =$ ____________ mℓ

$C_w =$ ____________ μg/mℓ

$C_1 =$ ____________ μg/mℓ

$V_a =$ ____________ mℓ

$V_w =$ ____________ mℓ

$C_a =$ ____________ μg/mℓ

(9) TOTAL BERYLLIUM EMISSIONS:

$$R = \frac{.0864\ W_t\ (v_s)_{avg}\ A_s}{V_{total}}$$

where

$W_t =$ ____________ μg

$(v_s)_{avg} =$ ____________ m/sec

$V_{total} =$ ____________ m^3

$A_s =$ ____________ m^2

(10) ISOKINETIC VARIATION:

$$I = \frac{100\ V_{total}}{A_n\ \theta (v_s)_{avg}} =$$ ____________ percent

where

$V_{total} =$ ____________ m^3

$\theta =$ ____________ sec

$A_n =$ ____________ m^2

$(v_s)_{avg} =$ ____________ m/sec

Figure 6. Sample calculation and data analysis form (cont.)

$K_w = 0.00346 \frac{\text{mm Hg - m}^3}{\text{m}\ell\ {}^\circ\text{K}}$

$= \left(0.00267 \frac{\text{in Hg - ft}^3}{\text{m}\ell\ {}^\circ\text{R}}\right)$ when these units are used

V_{1_c} = Total volume of liquid collected in impingers and silica gel, mℓ

T_s = Average stack gas temperature, °K (°R)

P_s = Stack pressure, mm Hg (in. Hg)

The stack pressure is determined by:

$P_s = P_{bar} \pm$ Static pressure

2.5.3.2 Sample Dry Gas Volume, V_{m_s}, Correct the sample volume measured by the dry gas meter to stack conditions using this equation:

$$V_{m_s} = V_m \frac{T_s}{T_m} \left(\frac{P_{bar} + \frac{\Delta H_{avg}}{13.6}}{P_s}\right) \qquad (3)$$

where

V_{m_s} = Volume of gas sample through the dry gas meter at stack conditions, m^3 (ft^3)

V_m = Volume of gas sample through the dry gas meter at meter conditions, m^3 (ft^3)

T_s = Average temperature of stack gas, °K(°R)

T_m = Average dry gas meter temperature, °K(°R)

P_{bar} = Barometric pressure at the orifice meter, mm Hg (in. Hg)

ΔH_{avg} = Average pressure drop across the orifice meter, mm H_2O (in. H_2O)

13.6 = Specific gravity of mercury

P_s = Stack pressure, $P_{bar} \pm$ static pressure, mm Hg (in. Hg)

Temperatures are converted to degrees Kelvin (Rankine), and all pressures are recorded to the nearest 2.5 mm Hg (0.1 in. Hg). Average the dry gas meter temperatures of the $[(T_{m_{in}})°C]$ and $[(T_{m_{out}})°C]$ to obtain the average temperature of the gas flowing through the meter during the test. Average the pressure of the stack (P_s). Determine the average orifice pressure drop by totaling the ΔH values at each traverse point and dividing by the total number of traverse points. Orifice pressure readings and the calculated average in millimeters of water should be rounded to two significant digits (e.g., 0.12 or 1.2). Record V_{m_s} to the nearest .003 m^3 (0.1 ft^3).

2.5.3.3 Total Gas Volume, V_{total}. Use equation 104-4 (from app. A) to calculate the total gas volume where:

$$V_{total} = V_{m_s} + V_{w_s} \qquad (4)$$

where

V_{total} = Total volume of gas sample at stack conditions, m^3 (ft^3)
V_{m_s} = Volume of gas through dry gas meter at stack conditions, m^3 (ft^3)
V_{w_s} = Volume of water vapor in gas sample at stack conditions, m^3 (ft^3) (See subsection 2.5.3.2)

2.5.3.4 Stack Gas Molecular Weight on a Wet Basis, M_s. Calculate the stack gas molecular weight by

$$M_s = M_d\ (1 - B_{wo}) + 18B_{wo} \qquad (5)$$

where M_d is given by

$$M_d = 0.44(\%\overline{CO_2}) + 0.32(\%\overline{O_2}) + 0.28(100 - \%\overline{CO_2} - \%\overline{O_2}) \qquad (6)$$

and $\%\overline{CO_2}$ and $\%\overline{O_2}$ are the averages of percent CO_2 and O_2 determinations, respectively, according to the Quality Assurance Document of this series for Method 3, (ref. 7).

Record M_s to three significant digits (i.e., --.-) on the data sheet of figure 6.

2.5.3.5 Average Stack Gas Velocity $(v_s)_{avg}$. Use the following equation to calculate the average stack gas velocity (ref. 5):

$$(v_s)_{avg} = K_p C_p (\sqrt{\Delta P})_{avg} \sqrt{\frac{(T_s)_{avg}}{P_s M_s}} \qquad (7)$$

where

$(v_s)_{avg}$ = Average stack gas velocity, m/s (ft/s)

$K_p = 34.98 \ \frac{m}{sec} \left(\frac{g\text{-mm Hg}}{g\text{-mole } ^\circ K \text{ mm } H_2O} \right)$

$= \left(85.53 \ \frac{ft}{sec} \left(\frac{lb\text{-in Hg}}{lb\text{-mole } ^\circ R \text{ in. } H_2O} \right)^{1/2} \right)$

when these units are used

C_p = Pitot tube coefficient, dimensionless

$(T_s)_{avg}$ = Average stack gas temperature, °K (°R)

$(\sqrt{\Delta P})_{avg}$ = Average square root of the velocity head of stack gas, $\sqrt{\text{mm Hg}}$ (in. $H_2O)^{1/2}$ (see figure 5)

P_s = Stack pressure, $P_{bar} \pm$ static pressure, mm Hg (in. Hg)

M_s = Molecular weight of stack gas on a wet basis, the summation of the products of the molecular weight of each component multiplied by its volumetric proportion in the mixture, g/g mole (lb/lb-mole) [2.5.3.4].

2.5.3.6 Total Beryllium Collected, W_t. Determine the total weight of beryllium collected using this equation:

$$W_t = V_l C_l - V_w C_w - V_a C_a \qquad (8)$$

where

W_t - Total collected weight of beryllium, μg

V_l = Total volume of hydrochloric acid (from subsec. 2.5.2.6(2)), mℓ.

C_l = Concentration of beryllium found in sample, μg/mℓ.

V_w = Total volume of water used in sampling (impinger contents plus all wash amounts), mℓ.

C_w = Blank concentration of beryllium in water, μg/mℓ

V_a = Total volume of acetone used in sampling (all wash amounts), mℓ

C_a = Blank concentration of beryllium in acetone, μg/mℓ

2.5.3.7 Total Beryllium Emissions. Calculate the total beryllium emissions from each stack per day by utilizing the following equation which applies to a continuous operation. For operations that are cyclic in nature, use only the time per day that each stack is in operation. The total emissions from a source will be the summation of results from all stacks.

$$R = \frac{W_t \, (v_s)_{avg} \, A_s}{V_{total}} \times \frac{86{,}400 \text{ s/d}}{10^6 \text{ μg/g}} \qquad (9)$$

where

R = Rate of emission, g/d

W_t = Total weight of beryllium collected, μg (from subsec. 2.5.3.6)

V_{total} = Total volume of gas sample at stack conditions, m^3 (ft^3) (from subsec. 2.5.3.3)

$(v_s)_{avg}$ = Average stack gas velocity, m/s (ft/s) (from subsec. 2.5.3.5)

A_s = Stack area, m^2 (ft^2)

86,400 = Number of seconds per day

2.5.3.8 Isokinetic Variation. The comparison of the velocity of gas in the probe tip versus the actual stack gas velocity is calculated using equation 104-8 (from app. A).

$$I = \frac{100\ V_{total}}{A_N\ \theta (v_s)_{avg}} \tag{10}$$

where

I = Percent of isokinetic sampling

V_{total} = Total volume of gas sample at stack conditions, m^3 (ft^3) (from subsec. 2.5.3.3)

A_N = Probe tip area, m^2 (ft^2)

θ = Sampling time, s

$(v_s)_{avg}$ = Average stack gas velocity, m/s (ft/s) (from subsec. 2.5.3.5)

SECTION III MANUAL FOR FIELD TEAM SUPERVISOR

3.0 GENERAL

The term "supervisor" as used in this document applies to the individual in charge of a field team. He is directly responsible for the validity and the quality of the field data collected by his team. He may be a member of an organization which performs source sampling under contract to government or industry, a government agency performing source sampling, or an industry performing its own source sampling activities.

It is the responsibility of the supervisor to identify sources of uncertainty or error in the measurement process for specified situations and, if possible, eliminate or minimize them by applying appropriate quality control procedures to assure that the data collected are of acceptable quality. Specific actions and operations required of the supervisor for a viable quality assurance program are summarized in the following listing.

1. Monitor/Control Data Quality

    a. Direct the field team in performing field tests according to the procedures given in the Operations Manual.

    b. Perform or qualify results of the quality control checks (i.e., assure that checks are valid).

    c. Perform necessary calculations and compare quality control checks to suggested performance criteria.

    d. Make corrections or alter operations when suggested performance criteria are exceeded.

    e. Forward qualified data for additional internal review or to user.

2. Evaluate Routine Operation

    a. Obtain from team members immediate reports of suspicious data or malfunctions. Initiate corrective action or, if necessary, specify special checks to determine the trouble; then take corrective action. Document the corrective action taken.

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b. Examine the team's log books periodically for completeness and adherence to operating procedures.

c. Approve data sheets, calibration checks, etc., for filing.

3. Evaluate Overall System

a. Evaluate available alternative(s) for accomplishing a given objective in light of experience and needs,

b. Evaluate operator training/instructional needs for specific operations.

Consistent with the realization of the objectives of a quality assurance program as given in section I, this section provides the supervisor with brief guidelines and directions for:

1. Collection of information necessary for assessing data quality on an intrateam basis;

2. The use of performance criteria to insure the collection of data of acceptable precision/accuracy;

3. Isolation, evaluation, and monitoring of major components of system variability.

In subsection 3.1, a method of assessing data quality on an intrateam basis is given. This method involves calculating a sample standard deviation using the three replicate runs required in a field test and calculating 90 percent confidence limits for the average of the three replicates.

Subsection 3.2 presents suggested criteria for judging equipment performance, frequency of calibration, and isokinetic sampling.

Directions for the collection and analysis of information to identify trouble, and subsequently, the control of data quality within acceptable limits are given in the third subsection.

3.1 ASSESSMENT OF DATA

The beryllium emission rate, $\overline{R}$, for a particular field test is the average of at least three replicates. Intrateam assessment of data quality as discussed herein provides for an estimate of the precision of the measurements. Precision in this case refers to replicability, i.e., the variability among replicates and is expressed as a coefficient of variation. This precision statement combines variability due to process changes and to measurement errors. This technique does not provide the information necessary for estimating measurement bias (see subsection 4.1.2 for a discussion of bias) that could occur, for example, from an error in determining the pitot tube coefficient, nozzle cross-sectional area, or the orifice meter calibration. However, if the operating procedures given in the Operations Manual are followed, the bias should be small in most cases. An independent quality audit which would make possible a bias estimate is suggested and discussed in section IV, the Management Manual.

3.1.1 Calculating Precision of Field Data

Each field test is comprised of at least three sample runs. Using the sample runs as replicates, a standard deviation can be calculated. This calculated standard deviation is a combined measure of the measurement and process variabilities. The standard deviation is calculated by

$$s\{R\} = \left[\frac{(R_1 - \bar{R})^2 + (R_2 - \bar{R})^2 + (R_3 - \bar{R})^2}{2}\right]^{1/2} \qquad (11)$$

where $s\{R\}$ = The calculated standard deviation for the three sample runs, g/d,

$R_1(R_2)(R_3)$ = Beryllium emission rate for sample run 1 (2)(3), g/d,

$\bar{R}$ = Average beryllium emission rate calculated from the three sample runs, i.e., $1/3(R_1 + R_2 + R_3)$, g/d, and

2 = The number of replicates minus one (degrees of freedom).

3.1.2 Reporting Data Quality

It is recommended that the average measured beryllium emission rate, $\bar{R}$, be reported with 90 percent confidence limits. Assuming that $\bar{R}$ is normally distributed (this is usually a valid assumption since sample means tend to be normally distributed even for non-normal parent distributions) and using $s\{R\}$ as calculated in 3.1.1 above to estimate the standard deviation, exact confidence limits can be calculated for the true R value using the Student t-distribution with $r - 1 = 2$ degrees of freedom. This assumes no bias in the average values. The average measured value with 90 percent confidence limits is reported as

$$\bar{R} \pm 2.92\ s\{R\}/\sqrt{n} \tag{12}$$

where $\bar{R}$ = The average of three replicates, g/d.

$s\{R\}$ = Estimated standard deviation of R based on three replicates, g/d.

2.92 = 95th percentile of the Student t-distribution with 2 degrees of freedom which yields a 90 percent confidence interval.

n = The number of replicates, i.e., $n = 3$ for this case.

For example, if for a given field test $\bar{R} = 8.52$ g/d and $s\{R\}$ was calculated to be .49 g/d the reported value with 90 percent confidence limits would be

$$8.52\ \text{g/d} \pm (2.92)(.49\ \text{g/d}) \Big/ \sqrt{3}$$

or the true beryllium emission rate, $\bar{R}_t$, would be assumed to be in the interval

$$7.69\ \text{g/d} \le \bar{R}_t \le 9.35\ \text{g/d}$$

The utility of the above statement follows from the fact that if this procedure for computing confidence limits is followed for several field tests, then 90 percent of the time the true $\bar{R}_t$ value will be contained within the given limits (assuming that $\bar{R}$ is not biased). It is recommended that the 90 percent confidence limits be reported with the field data form in the Operations Manual.

3.2 SUGGESTED PERFORMANCE CRITERIA

Data assessment as discussed in the previous subsection is based on the premise that all variables are controlled within a given level, thereby guarding against large undetected biases in the measurement process. These levels of suggested performance criteria are the values given in the Operations Manual for determining when equipment and/or personnel variability is out of control. Criteria for judging performance are summarized in table 1.

3.3 COLLECTION AND ANALYSIS OF INFORMATION TO IDENTIFY TROUBLE

In a quality assurance program, one of the most effective means of preventing trouble is to respond immediately to indications of suspicious data or equipment malfunctions. There are certain visual and operational checks that can be performed while the measurements are being made to help insure the collection of data of good quality. These checks are written as part of the routine operating procedures in section II. In order to effectively apply preventive-type maintenance procedures to the measurement process, the supervisor must know the important variables in the process, know how to monitor the critical variables, and know how to interpret the data obtained from monitoring operations. These subjects are discussed in the following subsections.

3.3.1 Identification of Important Variables

Determination of the beryllium emission rate requires a sequence of operations and measurements that yields as an end result a number that serves to represent the average beryllium emission rate for that field test. There is no way of knowing the accuracy, i.e., the agreement between the measured and the true value, for a given field test. However, a

Table 1. Suggested Performance Criteria

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1. Suggested Criteria for Equipment Performance

(a)	Dry Gas Meter:	$.98 \leq \gamma \leq 1.02$
(b)	Barometer:	± 5.08 mm Hg (± 0.2 in. Hg)
(c)	Thermometers:	± 2.8°K (± 5°R) at 273°K (492°R) or ± 3.9°K (± 7°R) at 373°K (672°R)
(d)	Stack Temperature Measuring System:	± 2.8°K (± 5°R) at 273°K (492°R) or ± 3.9°K (± 7°R) at 373°K (672°R)
(e)	Sampling Train Leakage:	Less than .00057 m^3/min (0.02 ft^3/min) at 381 mm Hg (15 in. Hg) vacuum
(f)	Meter Orifice:	$\Delta H@$ constant within 3.81 mm H_2O (± 0.15 in. H_2O) over the operating range.
(g)	Probe Nozzle Diameter:	Range of three different diameter measurements less than .010 cm (0.004 in.).
(h)	Type-S Pitot Tube:	C_p constant within ± 5 percent over working range and each calibration check is within 1.2 percent of the original C_p.

2. Suggested Criteria for Performing Equipment Calibration

(a) Above items (a) through (g) are calibrated when new and checked before each field test and recalibrated any time the check results fall outside the prescribed performance limits.

(b) Item (h) the type-S pitot tube is calibrated when new, before every third field test, or at any sign of damage.

3. Suggested Criteria for Percent Isokinetic

$$0.90 \leq I \leq 1.10$$

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knowledge of the important variables and their characteristics allows for the application of quality control procedures to control the effect of each variable at a given level during the field test, thus providing a certain degree of confidence in the validity of the final result.

A functional analysis of this method of measuring the beryllium emission rate of a stationary source was made to try to identify important components of system error. The method has been subjected to one collaborative test (ref 16). The results from this test were used, along with results from an evaluation study of Method 5 and (ref. 17), to estimate the overall system error. The use of the Method 5 evaluation is valid because the identical field procedures are used for Method 104. Individual error components were estimated using engineering judgment in a manner such that their combined variability is consistent with overall system error.

Variability in emissions data derived from multiple repetitions include components of variation from:

1. Process conditions,
2. Equipment and personnel in field procedures, and
3. Equipment and personnel in the laboratory.

In many instances time variations in source output may be the most significant factor in the total variability. In order to judge the relative magnitudes of measurement variability and process output variability, process parameters should be monitored throughout the test. The exact process data to be obtained are dependent upon the process being tested. In general, all factors which have a bearing on the emissions should be recorded on approximately a 15-minute interval (ref. 18). These factors include process or fuel weight rate, production rate, temperature and pressure in the reactor and/or boiler, control equipment, fan and/or damper settings, pressure drop or other indicator of beryllium collection efficiency. Sample forms for combustion, incineration, and process sources are given in reference 18.

It is most important to realize that the larger measurement errors result from poor operator technique such as loss or gain of collected beryllium during sample recovery, or poor orientation and positioning of the probe during sample collection. Such deviations from recommended procedures cannot be evaluated or corrected for. It is important to observe and eliminate such occurrences while the test is in process.

Sources of variation due to equipment includes the type-S pitot tube, sampling nozzle cross-sectional area, orifice meter, dry gas meter, probe heater, and sample box heater. These are all controlled through a calibration check before each field test. Also, the probe and sample box heaters are checked periodically during the test. Important error sources checked immediately before and/or during sample collection includes sampling train leaks, the sample gas temperature leaving the last impinger, and isokinetic sampling conditions. Assuming good operator technique, these error sources are discussed below and each one's effect on the determination of the beryllium emission rate is derived from a functional analysis of the measurement process in subsection 4.1.

The analysis phase of this method is also subject to large variability if proper care is not exercised. All discussions of the analysis phase assume that the analysis is made by atomic absorption. The precision and accuracy of the data obtained from this method of analysis depend upon equipment performance and the proficiency and conscientiousness of the analyst performing the task.

A summary of the important parameters is given below. The parameters are roughly given in ascending order of importance. Importance is qualitatively derived from the estimated error range of a parameter weighted by its estimated frequency of occurrence.

3.3.1.1 Equipment Calibration. Equipment calibration is the backbone of any quality assurance program. It is important that the calibration procedure be carried out correctly, that the calibration standards are properly calibrated and maintained, and that the frequency of calibration is adequate.

Important calibration constants and how they influence measurement accuracy include the following:

1. Error in the pitot tube calibration coefficient is directly reflected in the stack gas velocity determination and is doubled in the process of determining isokinetic sampling rates. This error could be very large if the pitot tube is not calibrated under actual field test conditions, i.e., strapped to the sampling probe with isokinetic sampling conditions maintained at each calibration point.

2. Error in determining the nozzle diameter is quadrupled in the process of determining isokinetic sampling rates and is doubled in the percent of isokinetic sampling calculation. One source of error here is the use of an out-of-round nozzle. The average nozzle diameter is used to calculate the area of a circle which yields a larger than true cross-sectional area if the nozzle tip is not round.

3. Dry gas meter inaccuracy appears directly in the concentration and beryllium emission rate determinations.

4. The orifice meter calibration constant is used in determining isokinetic sampling conditions and any error in the constant is doubled in setting the sampling rate. Also, if ΔH@, the pressure drop across the orifice that gives a flow rate of .021 m^3/min (0.75 ft^3/min) at 21°C (70°F) and 760 mm Hg (29.92 in. Hg) varies from 46.7 mm H_2O (1.84 in. H_2O), and a nomograph is used to set isokinetic sampling conditions, an error results. It is recommended that an orifice meter with a ΔH@ outside the range of 40.4 to 53.1 mm H_2O (1.59 to 2.09 in. H_2O) not be used in conjunction with a nomograph.

3.3.1.2 Anisokinetic Sampling. Anisokinetic sampling can occur from error in the calibration constants of the pitot tube, orifice meter, and nozzle diameter. It can also result to a lesser degree, usually, from measurement error in the moisture content and molecular weight of the stack gas. Errors from the above sources will not be directly reflected in the percent of isokinetic sampling calculation. Therefore, it is important to determine each parameter as accurately as possible, either through calibrations or careful measurements.

Failure or, in some instances, the inability to make adjustments in the sampling rate as the stack gas velocity varies or as the deposited particulate matter plugs the filter can result in anisokinetic sampling. Use of a nomograph can be a cause of anisokinetic sampling because of (1) any inaccuracy in the nomograph, (2) use of preset values for C_p, $\Delta H@$, and M_d (these errors can be eliminated by using actual values and adjusting the correction factor on the nomograph), and (3) operator error in setting the nomograph. The sum of these errors is quantified to a certain extent by the percent of isokinetic sampling calculation.

Deviation from isokinetic sampling cannot be related directly to error in the measurement process (see subsection 4.1). However, failure to maintain isokinetic sampling conditions under otherwise normal operations reflects the lack of alertness and, perhaps even the level of competency, of the field crew.

3.3.1.3 Sample Recovery. The technique used by, and the attitude of, the crew members in sample recovery are of paramount importance to measurement precision and accuracy. Use of an inadequate sample recovery area in terms of space, lighting, or cleanliness will increase the probability of error. Results from a collaborative test (ref. 16) have shown that the recovery of beryllium-containing particles with the washing of the probe, filter holder, and impingers is a major source of error when compared to the filter-only collection. An estimate (ref. 16) of 77 percent of the total beryllium collected is from the wash solution in the second sample container (section 2.4.4.2), hence contamination of the wash solution, failure to properly clean all of the sampling equipment, or loss of wash solution could cause considerable error in the final measurement value.

3.3.1.4 Analysis. The analysis phase is subject to error from any of the following:

1. Sample handling. Exposure to the atmosphere at any time between sample collection and analysis could result in contamination or

loss of the sample. Also, insolubles that penetrate the filter and are caught in the impingers may be lost in transfers to and from container 2 due to the non-homogeneity of the sample.

2. Digestion. Failure to thoroughly dissolve any of the particles from either of the two sample containers in the solution will be a source of negative error in the final measurement value. Stated simply, the large beryllium containing particles will not absorb the 234.8 nm wavelength of energy.
3. Preparation of standards. Preparation of standards for a working analytical curve can be a source of errors if proper techniques are not followed and care taken in the process.
4. Setting and maintaining standard conditions throughout the analysis period. Errors can arise from improper instrument tuning, a ragged burner gas flame, and an incorrect aspiration rate. Measurement of working standard solutions before and after field sample analysis, blank samples measured before every field sample and methods for eliminating interferences from other metals are used to ensure data of acceptable quality from the analysis phase of the measurement method.

3.3.1.5 Calculations. Calculations for this method are known to be a major source of error. Some calculations involve several terms and should only be attempted (for the final report) at a desk or work table and preferably with the aid of a calculator or at least a good slide rule. A computer program using raw data as an input is highly recommended for making the final calculations.

As a check, it is recommended that all calculations be independently repeated from raw data.

3.3.2 How to Monitor Important Variables

In general, if the procedures outlined in the Operations Manual are followed, the major sources of measurement variability will be in control. It is felt, however, that the supervisor should visually check certain parameters and operations periodically while measurements are being made to insure good operator technique and the proper use of equipment. The parameters and operations to check are essentially those recommended for the auditor as listed in subsection 4.3.

Results of the calibration checks for the dry gas meter, orifice meter, nozzle diameter, and pitot tube should be checked before each field test. Any item of equipment not satisfying the suggested performance criteria of table 1 should be calibrated or replaced.

There appears to be a need for actual field data on several of the parameters or variables involved in this measurement method in order to better judge their influence on measurement variability. One of the most effective means of identifying and quantifying important sources of variability is through the use of quality control charts. Quality control charts will provide a basis for action with regard to the measurement process; namely, whether the process is satisfactory and should be left alone, or whether the process is out of control and action should be taken to find and eliminate the causes of excess variability. In the case of this method in which documented precision data are scarce, the quality control charts can be evaluated after a period of time to determine the range of variation that can be expected under normal operating conditions. Also, even though results from individual field tests are within bounds, trends can be identified and corrective action taken further improving data quality through the proper use of control charts.

Discussions of control charts and instructions for constructing and maintaining them are given in many textbooks in statistics and quality control, such as in references 19 and 20.

It is good practice to note directly on control charts the reason for out-of-control conditions, if determined, and the corrective actions taken. It is also good practice to maintain control charts in large size, e.g., 8-1/2 x 11 (inches) or larger and to keep them posted on a wall for viewing by all concerned, rather than have them filed in a notebook. Recommended control charts are discussed below.

3.3.2.1 Pitot Tube Calibration Coefficient. A sample control chart for pitot tube calibration checks is given in the quality assurance document of this series for Method 2--Determination of Stack Gas Velocity and Volumetric Flow Rate (type-S pitot tube)--(ref. 3), page 38.

3.3.2.2 Range Chart for Beryllium Emission Rate Replicates. In compliance testing where it is desired to determine the source output at a fixed level of operation, a large range in the three replicates would suggest process variability and/or measurement variability. Expressing the range, RG, as a percent of the average, i.e., the difference in the largest and smallest of the three replicates divided by the average of the three replicates, all multiplied by 100; a control chart with limits as given in figure 7 can be used initially. These limits are based on the assumption of equal variability in the source output and the measurement process. (As field data becomes available more appropriate estimates can be made.) Therefore, the coefficient of variation used in constructing the control chart becomes $\sqrt{2} \times CV\{R\} = \sqrt{2} \times 37 = 52.$* The upper control limit (UCL) is calculated using the following equation:

$$UCL = D_2 \; CV = D_2 \times \sqrt{2} \; CV\{R\} \qquad (13)$$

where D_2 is the factor for three-sigma control limits (3σ) for range values when standards are given (ref. 21). Table 2 lists several D_2 values as a function of the number of samples. Determining limits in this way serves as a starting point in the analysis of the data. When a data point falls out of bounds on this graph, the process data should be checked to see if the process changed between runs and the percent of isokinetic calculations checked to see if one run was significantly different from the others in order to identify the cause of the excessive variability. Note then that exceeding the upper control limit does not necessarily invalidate the test data.

3.3.2.3 Mean and Range Charts for Percent of Isokinetic Sampling. Maintaining isokinetic sampling conditions is important in beryllium sampling. Control charts displaying the range and mean of percent isokinetic sampling provide, at a glance, means for evaluating the performance of a team or groups of teams over an extended period of time. If deviations from

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* See section 4.1.4 for CV{R} = 27.

Table 2. Factors for Control Charts for the Range, RG

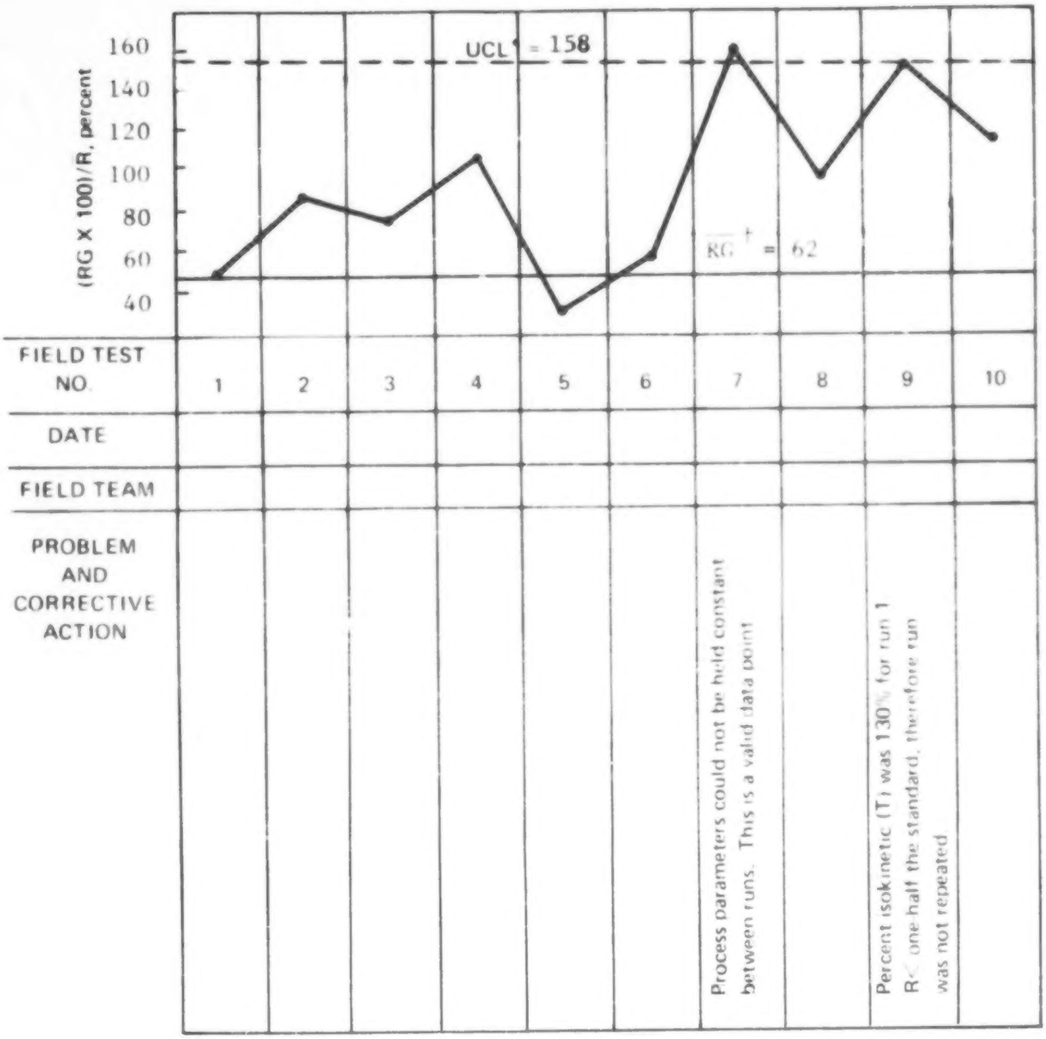
Number of Samples n	Factor for Center Line d^2	Factor for Control Limits	
		Lower D_1	Upper D_2
2	1.128	0	3.686
3	1.693	0.3	4.358
4	2.059	0	4.698
5	2.326	0	4.918
6	2.534	0	5.078
7	2.704	0.205	5.203
8	2.847	0.387	5.307
9	2.970	0.546	5.394
10	3.078	0.687	5.469
11	3.173	0.812	5.534
12	3.258	0.924	5.592
13	3.336	1.026	5.646
14	3.407	1.121	5.693
15	3.472	1.207	5.737

isokinetic greater than ± 10 percent are not allowed, i.e., the run has to be repeated, then in a rough way 10 percent can be taken as the 3σ value giving a standard deviation of about 3.3 percent. Based on three replicates and the above standard deviation, the range chart and mean charts are given in figures 8 and 9, respectively.

The RG values are plotted sequentially as they are obtained and connected to the previously plotted point with a straight line. Corrective action, such as instruction in proper operating technique, should be taken before the next field test any time one of the following criteria are exceeded:

1. One point falls outside the UCL.
2. Seven consecutive points fall above the $\overline{RG}$ line.

Exceeding the first criteria will usually indicate poor technique or equipment malfunction between sample runs of a particular field test. Exceeding the second criteria indicates a systematic error due to equipment



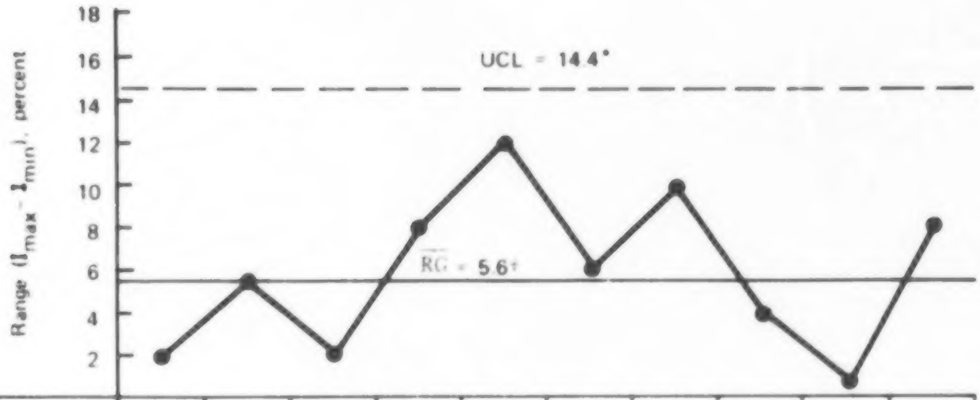
Process parameters could not be held constant between runs. This is a valid data point

Percent isokinetic (T) was 130% for run 1 R< one half the standard, therefore run was not repeated

*UCL = $D_2 CV = 4.358 \times 37 \times \sqrt{2} = 158.4$

$^{\dagger}\bar{RG} = d_2 CV = 1.693 \times 25.7 \times \sqrt{2} = 43.5$

Figure 7. Sample control for the range, RG of R replicates.

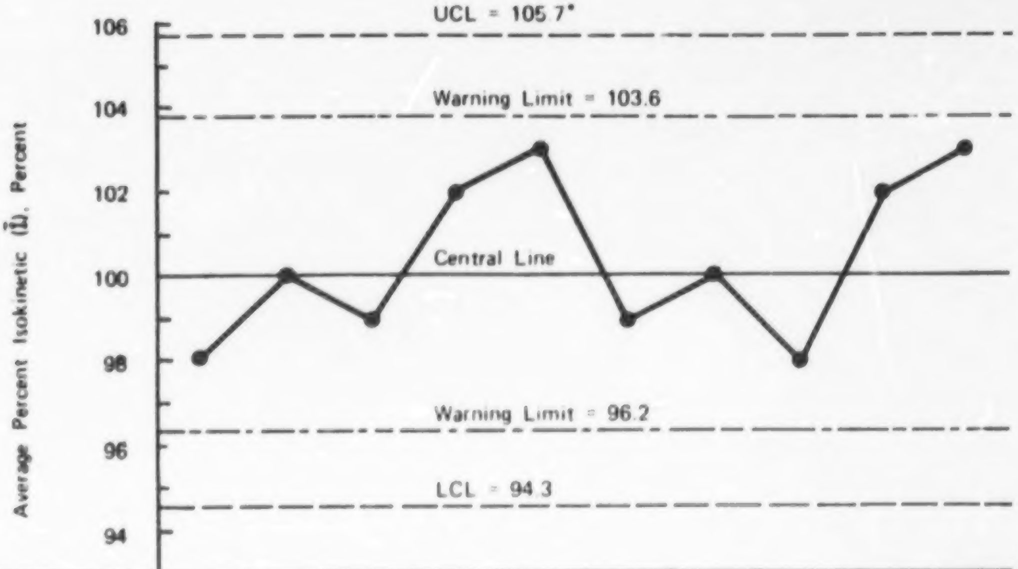


FIELD TEST NO.	1	2	3	4	5	6	7	8	9	10
DATE										
FIELD TEAM										
PROBLEM AND CORRECTION ACTION										

*UCL = $D_2\sigma$ = 4.358 × 3.3 = 14.4

†$\overline{RG}$ = $d_2\sigma$ = 1.693 × 3.3 = 5.6

Figure 8. Sample control chart for the range, RG of percent isokinetic, I, sampling for three test runs per field test.



FIELD TEST NO.	1	2	3	4	5	6	7	8	9	10
DATE										
FIELD TEAM										
PROBLEM AND CORRECTIVE ACTION										

*UCL = $\bar{I} + 3\sigma(\bar{I}) = 100 + 3 \times 3.3/\sqrt{3} = 105.7$

Figure 9. Sample control chart for the average percent of isokinetic sampling per field test.

bias or poor technique. (Note that the UCL can be exceeded without violating the I = 100 ± 10 interval for any one run.)

The $\overline{I}$ values, i.e., average percent of isokinetic sampling per three sampling runs, are plotted sequentially as they are obtained from field tests and connected to the previously plotted point with a straight line. Corrective action, such as instruction in proper operating technique and/or performing equipment calibration checks should be taken before attempting the next field test any time one of the following criteria is exceeded:

1. One point falls outside the UCL or LCL.

2. Two out of three consecutive points fall in the warning zone (between 2σ and 3σ limits).

3. Seven consecutive points fall on the same side of the center line.

Exceeding the first criteria will usually indicate poor technique or equipment malfunction. The second and third criterion when exceeded indicate an assignable source of variability due either to faulty equipment or a consistent error in performing the operation procedures.

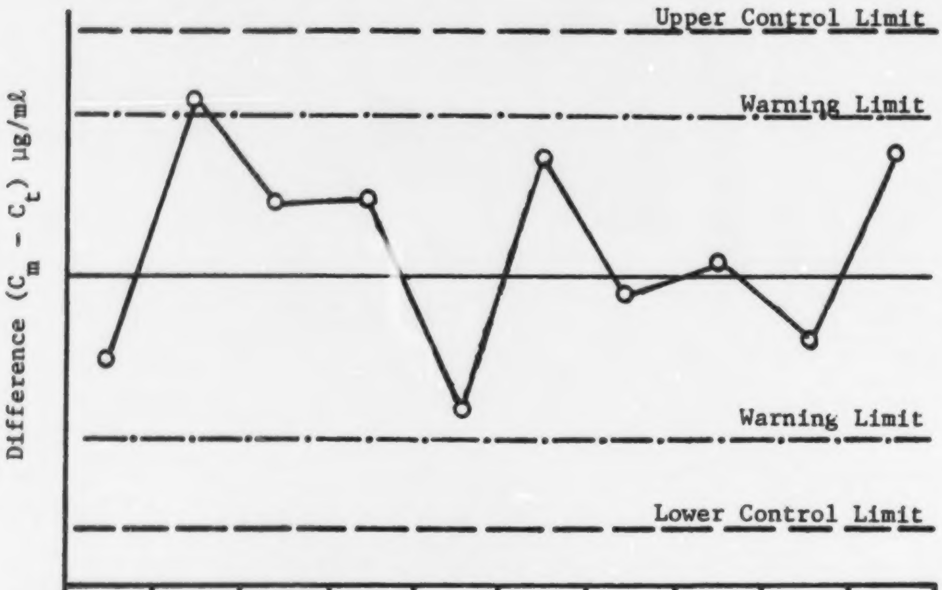
3.3.2.4 Control Chart for the Measurement of Working Standard Solutions. Frequent measurement of working standard solutions is the best means of assuring stability of the spectrometer during sample analysis. It is assumed herein that deviations greater than ± 0.18 μg/mℓ from the known value are sufficient to require the development of a new calibration curve. Assuming that the difference in the measured (C_m) and known concentrations (C_t) is normally distributed about a zero mean with a standard deviation of 0.6 μg/mℓ, a control chart such as shown in figure 10 can be used.

Corrective action such as generating a new calibration curve should be taken any time one of the following criteria is exceeded:

1. One point falls outside the UCL or LCL.

2. Two out of three consecutive points fall in the warning zone (between the 2σ and 3σ limits).

3. Seven consecutive points fall on the same side of the center line.



FIELD TEST NO.	1	2	3	4	5	6	7	8	9	10
DATE										
FIELD TEAM										
PROBLEM AND CORRECTIVE ACTION										

Figure 10. Sample control chart for the measurement of working standard solutions.

SECTION IV MANUAL FOR MANAGER OF GROUPS OF FIELD TEAMS

4.0 GENERAL

The guidelines for managing quality assurance programs for use with Test Method 104 - Reference Method for Determination of Beryllium Emissions from Stationary Sources are given in this part of the field document. This information is written for the manager of several teams for measuring source emissions and for the appropriate EPA, State, or Federal Administrators of these programs. It is emphasized that if the analyst carefully adheres to the operational procedures and checks of Section II, then the errors and/or variations in the measured values should be consistent with the performance criteria as suggested. Consequently, the auditing routines given in this section provide a means of determining whether the stack sampling test teams of several organizations, agencies, or companies are following the suggested procedures. The audit function is primarily one of independently obtaining measurements and performing calculations where this can be done. The purpose of these guidelines is to:

1. Present information relative to the test method (a functional analysis) to identify the important operations and factors.

2. Present a methodology for comparing action options for improving the data quality and selecting the preferred action.

3. Present a data quality audit procedure for use in checking adherence to test methods and validating that performance criteria are being satisfied.

4. Present the statistical properties of the auditing procedure in order that the appropriate plan of action may be selected to yield an acceptable level of risk to be associated with the reported results.

These four purposes will be discussed in the order stated in the sections which follow. The first section will contain a functional analysis of the test method with the objectives of identifying the most important factors which affect the quality of the reported data and estimating

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the expected variation and bias in the measurements resulting from equipment and operator errors.

Section 4.2 contains several actions for improving the quality of the data; for example, by improved analysis techniques, instrumentation, and/or training programs. Each action is analyzed with respect to its potential improvement in the data quality as measured by its precision. These results are then compared on a cost basis to indicate how to select the preferred action. The cost estimates are used to illustrate the methodology. The manager or supervisor should supply his own cost data and his own actions for consideration. If it is decided not to conduct a data audit, sections 4.1 and 4.2 would still be appropriate as they contain a functional analysis of the reference method and of alternative methods or actions.

There are no absolute standards with which to compare the routinely derived measurements. Furthermore, the taking of completely independent measurements at the same time that the routine data are being collected (e.g., by introducing two sampling probes into the stack and collecting two samples simultaneously) is not considered practical due to the constrained environmental and space conditions under which the data are being collected. Hence, a combination of an on-site system audit, including visual observation of adherence to operating procedures, and quantitative performance audits is recommended as a dual means of independently checking on the source emissions data.

The third section contains a description of a data quality audit procedure. The most important variables identified in section 4.1 are considered in the audit. The procedure involves the random sampling of n stacks from a lot size of N = 20 stacks (or from the stacks to be tested during a three-month period, if less than 20) for which one firm is conducting the source emissions tests. For each of the stacks selected, independent measurements will be made of the indicated variables. These measurements will be used in conjunction with the routinely collected data to estimate the quality of the data being collected by the field teams.

The data quality audit procedure is an independent check of data collection and analysis techniques with respect to the important variables. It provides a means of assessing data collected by several teams and/or firms with the potential of identifying biases/excessive variation in the data collection procedures. A quality audit should not only provide an independent quality check, but also identify the weak points in the measurement process. Thus, the auditor, an individual chosen for his background knowledge of the measurement process, will be able to guide field teams in using improved techniques. In addition, the auditor is in a position to identify procedures employed by some field teams which are improvements over the current suggested ones, either in terms of data quality and/or time and cost of performance. The auditor's role will thus be one of aiding the quality control function for all field teams for which he is responsible, utilizing the cross-fertilization of good measurement techniques to improve the quality of the collected and reported data.

The statistical sampling and test procedure recommended is sampling by variables. This procedure is described in section 4.4. It makes maximum use of the data collected, and it is particularly adaptable to the small lot size and, consequently, the small sample size applications. The same sampling plans can be employed in the quality checks performed by a team or firm in its own operations. The objectives of the sampling and test procedure are to characterize data quality for the user and to identify potential sources of trouble in the data collection process for the purpose of correcting the deficiencies in data quality.

Section 4.4.4 describes how the level of auditing, i.e., the sample size n, may be determined on the basis of relative cost data and prior information about the data quality. This methodology is described in further detail in the Final Report on the Contract. The cost data and prior information concerning data quality are supplied to illustrate the procedure and these data must be supplied by the manager of groups of field teams depending upon the conditions particular to his responsibility.

Figure 11 provides an overall summary of the several aspects of the data quality assurance program as described in these documents. The flow diagram is subdivided into four areas by solid boundary lines. These areas correspond to specific sections or subsections of the document as indicated in the upper right hand corner of each area. The details are considered in these respective sections of the document and will not be described here.

4.1 FUNCTIONAL ANALYSIS OF TEST METHOD

Test Method 104 - Reference Method for Determination of Beryllium Emissions from Stationary Sources--as described in appendix A of this document is subjected to a functional analysis in this section. This method, in addition to its use in determining beryllium emissions, is also used to simultaneously determine the moisture content of the stack gas and to perform a velocity traverse from which the volumetric flow rate can be calculated. These results combined with the stack gas composition as measured by Method 3 yield a beryllium emission rate for the source being tested.

A functional analysis of the measurement process is performed for the following purposes:

1. To identify variables, operations, and factors that can influence the quality of the measurement data.
2. To determine how elements of uncertainty are propagated through the system to the resulting data.
3. To estimate the mean values and ranges of the various error sources existing under normal operating conditions and to determine the probable uncertainty to be associated with the reported measurements.

The results of the functional analysis provide information for the following:

1. Setting acceptable limits on data quality, i.e., precision and accuracy.

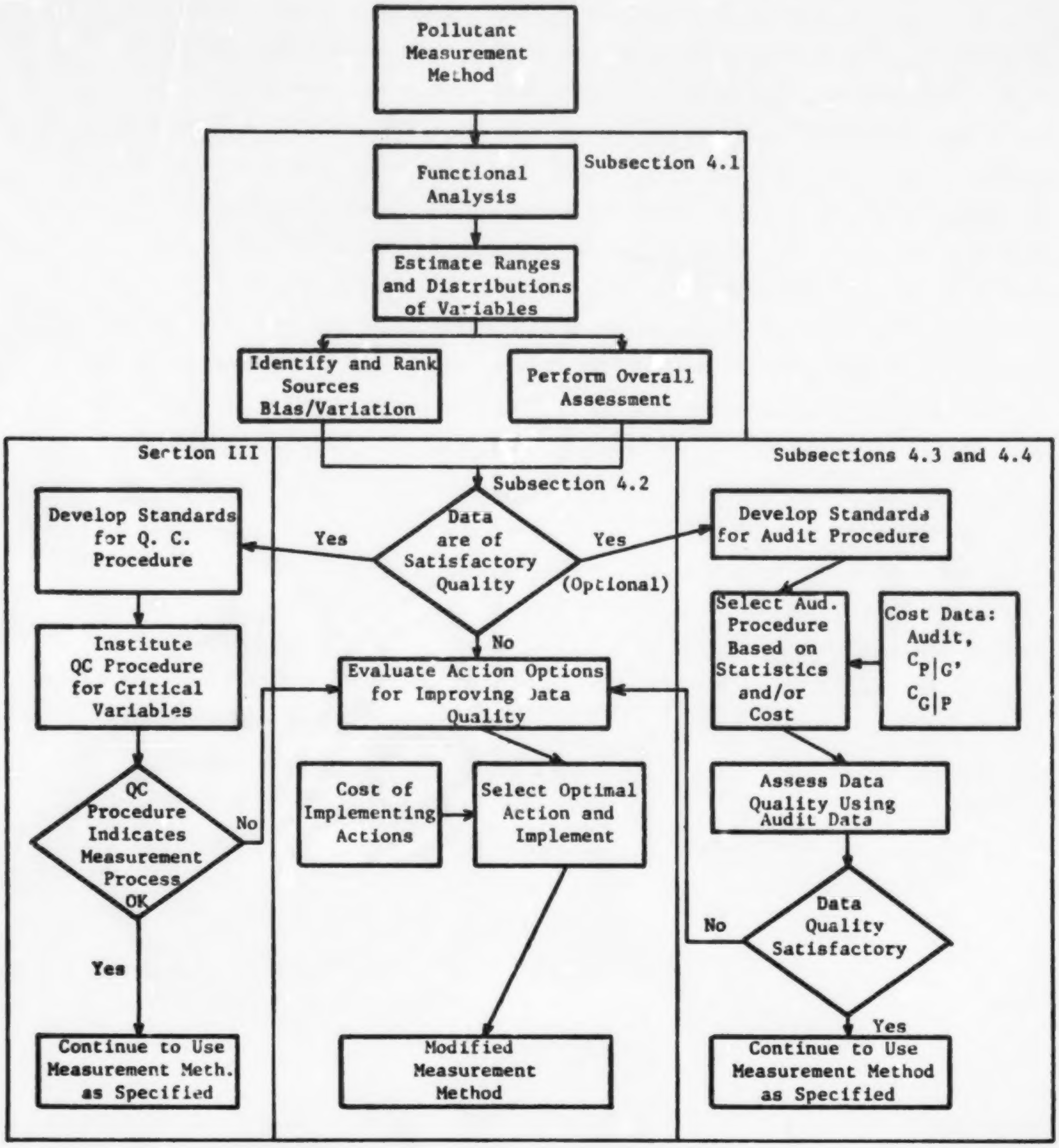

Pollutant Measurement Method
Functional Analysis
Subsection 4.1
Estimate Ranges and Distributions of Variables
Identify and Rank Sources Bias/Variation
Perform Overall Assessment
Section III
Subsection 4.2
Subsections 4.3 and 4.4
Develop Standards for Q. C. Procedure
Yes
Data are of Satisfactory Quality
Yes
(Optional)
Develop Standards for Audit Procedure
No
Institute QC Procedure for Critical Variables
Evaluate Action Options for Improving Data Quality
Select Aud. Procedure Based on Statistics and/or Cost
Cost Data: Audit, $C_{P|G}$, $C_{G|P}$
QC Procedure Indicates Measurement Process OK
No
Cost of Implementing Actions
Select Optimal Action and Implement
Assess Data Quality Using Audit Data
No
Data Quality Satisfactory
Yes
Yes
Continue to Use Measurement Meth. as Specified
Modified Measurement Method
Continue to Use Measurement Method as Specified

Figure 11. Summary of data quality assurance program.

2. Implementing quality controls to those factors that would otherwise result in excessive uncertainty in the resulting data.

3. Application of appropriate quality assurance practices (audits) at critical points in the measurement system with acceptable limits to be considered and actions to be taken when limits are exceeded.

This method for determining beryllium emission rates has been subjected to one collaborative test (ref. 16) hence some quantitative data is available. Except for the analysis phase, this method is almost identical in procedures and equipment to those required by Method 5 (ref. 16). For this reason, test data from the sampling phases of Method 5 is directly applicable to the same phases of Method 104 and has also been used in this document to depict representative accuracy and precision data. Estimates of the mean value and variability associated with the individual variables in the measuring rpocess were made using data from published documents. If data were not available, engineering judgments were used to estimate variable limits.

The functional analysis is discussed in two stages. Variable evaluation and range estimates are discussed first. For example, the mean value and variability on which the beryllium emission rate, R, depends will be given. Second, a variance analysis is performed to determine the sensitivity of the emission rate measurement to each of the variables. The results of this analysis are the basis for many of the limits used throughout this document.

The beryllium emission rate is calculated from the relationship:

$$R = K \times W_t \times \frac{Q_s}{V_{total}} \qquad (14)$$

where

R = Rate of beryllium emissions, g/d

$K = 8.6 \times 10^{-2} \frac{\text{g-s}}{\mu\text{g-d}}$

W_t = Total weight of beryllium collected, μg

Q_s = Volumetric flow rate of the stack gas, m^3/s

V_{total} = Total volume of gas sample at stack conditions, m^3.

The variability of the beryllium emission rate, R, will consist of components of variability from the individual determinations of W_t, Q_s, and V_{total}. Each of these three terms will be discussed separately below following a short definition of terms.

The variance analysis conducted in this section provides a statistical means of identifying important variables in the measurement process based on estimated variances and coefficients of variations. The mathematical basis for the procedures are described in the Final Report on Contract 68-02-0598 (ref. 22).

Many different measures of variability are conceivable depending upon the circumstances under which the measurements are performed. Only two extreme situations will be discussed here. They are:

1. Repeatability, is a measure of the variability in determinations made on the same sample (i.e., the same source with source parameters held as constant as possible) by the same field team using the same equipment over a short period of time. The repeatability standard deviation and the coefficient of variation are symbolized by σ_r and CV_r respectively.
2. Reproducibility is the variability associated with measurements made of the same sample (i.e., same source with source parameters held as constant as possible) by different field teams using different equipment over extended periods of time. The reproducibility standard deviation and the coefficient of variation are symbolized by σ_R and CV_R respectively.

The above definitions are based on a statistical model where each determination of the rate of beryllium emissions is the sum of three components as follows:

$$R = \overline{R} + b + e \qquad (15)$$

where R = the measured rate of beryllium emissions, g/d

$\overline{R}$ = the general average, rate of beryllium emissions, g/d

b = an error term representing the difference between laboratories, g/d

e = a random error occurring in each measurement, g/d.

In general, b can be considered as the sum:

$$b = b_r + b_s \tag{16}$$

where:

b_r = random component

b_s = systematic component

The term b is considered to be constant during any series of measurements performed under repeatable conditions, but to behave as a random variate in a series of measurements performed under reproducible conditions. Its variance is denoted by

$$\text{var } b = \sigma_L^2 \tag{17}$$

where σ_L^2 is the variance between laboratories or field teams, including the variance between analysts and between equipment.

The term e represents a random error occurring in each measurement. Its variance is denoted by

$$\text{var } e = \sigma_r^2, \tag{18}$$

where σ_r^2 is called the repeatability variance.

The reproducibility standard deviation is related to the between laboratories variance and the repeatability variance by

$$\sigma_R = \sqrt{\sigma_r^2 + \sigma_L^2} \,. \tag{19}$$

Repeatability and reproducibility of beryllium emission rate determinations will be discussed in the following subsections.

4.1.1 Weight of Beryllium Collected, W_t. The total collected beryllium, W_t, is given by

$$W_t = W_t' + \varepsilon \tag{20}$$

where W_t' represents the true beryllium mass per unit volume of stack gas at stack conditions and ε is the error associated with the collection and analysis of the beryllium sample. Important sources of error in the determination of W_t include:

1. Deviation from isokinetic sampling, (I);
2. Loss or gain of sample during sample recovery, (SR);
3. Volumetric measurement of collected sample, blank, and impinger contents plus wash amounts, (V); and
4. Variability in the spectrophotometric analysis phase of the measurement process, (A).

These four sources of error are independent; hence, the coefficient of variation of W_t, $CV\{W_t\}$, can be estimated by:

$$CV^2\{W_t\} = \frac{\sigma^2\{I\} + \sigma^2\{SR\} + \sigma^2\{V\} + \sigma^2\{A\}}{\bar{W}_t^2}$$

Using the ratios discussed in the following paragraphs, $CV^2\{W_t\}$ can be estimated to be .10. Therefore $CV\{W_t\} = 31$ percent.

4.1.1.1 Nonisokinetic sampling (I). Deviation from isokinetic sampling conditions can result in measurement error which is directly related to the degree of departure from isokinetic conditions and the particle size distribution in the sample gas. Departure from isokinetic sampling can occur, for instance, as a result of failure to adjust the nozzle velocity as the stack gas velocity varies. This type of departure will be detected when the percent of isokinetic sampling is calculated. Deviations from isokinetic sampling can also occur when errors are made in the pitot tube coefficient or in the nozzle diameter determinations. Unlike the example given above, these deviations from isokinetic sampling are not detectable from any checks that can be performed in the field

Sample gases containing particle size distributions extending into the 25 μm and above sizes are sensitive to nonisokinetic sampling error and each particle size distribution should be evaluated separately. In beryllium sampling the particle size is usually small; therefore, slight deviations from isokinesis (on the order of ± 10 percent from the acceptable range) will contribute negligibly to the overall system error. Based on this statement, then an estimated error in the beryllium emission rate determination due to nonisokinesis ($\sigma\{I\}/\bar{W}_t$) is .08.

4.1.1.2 Sample recovery (SR). A loss or gain of sample during sample recovery is mostly a function of technique and of the work area used for sample recovery. Sample recovery involves transferring the filter from the filter holder to a sample container and removing particulate matter lodged in the nozzle, probe, filter holder, and connecting glassware. No quantitative data are available for estimating this source of error. For this analysis, a coefficient of variation for repeatability of 2 percent is assumed. This, of course, indicates the best that can be achieved and does not include large deviations due to mistakes, accidents, or poor technique. A $\sigma\{SR\}/\bar{W}_t$ of .25 is assumed.

4.1.1.3 Volumetric measurement of collected sample, blank, and impinger contents,(V). The volumetric measurement error is a combination of three measurements as required by equation 7, section 2.5.3.6. Assuming that each volume is measured to the nearest mℓ, a $\sigma\{V\}/\bar{W}_t$ of .07 is reasonable as a result of the three independent measurements.

4.1.1.4 Analysis (A). Primary sources of variability in the analysis phase of the beryllium emission rate determination are discussed in subsection 3.3.1.4. They include:

1. Sample handling,
2. Preparation of standards, and
3. Setting and maintaining standard conditions throughout the analysis period.

Based on the analysis information acquired from reference 16, an estimated ratio of $\sigma\{A\}/\bar{W}_t$ of .15 is used in this document for illustrative purposes. This assumes that well-trained analysts make the determination in accordance with the recommendations in section 2 of this document in well-equipped laboratories.

4.1.2 **Volumetric Flow Rate, Q_s.** A functional analysis of the method used to measure volumetric flow rate was made in the quality assurance guidelines document for Method 2 (ref. 3). The results of that analysis will be used here without repeating the analysis itself. A repeatability coefficient of variation of 2.33 percent was derived for the volumetric flow rate. This value is used here as an estimate of how well a field team can repeat its measurement using the same equipment, assuming the velocity profile remains constant, and with only short periods of time between measurements. A higher value of 3 percent is used to represent the reproducibility coefficient of variation.

4.1.3 **Total Volume of Gas Sample at Stack Conditions, V_{total}.** The total gas sample volume is given by

$$V_{total} = V_{m_s} + V_{w_s} \qquad (22)$$

where

V_{total} = Total gas sample volume at stack conditions, m^3

V_{m_s} = Volume measured by the dry gas meter at stack conditions, m^3

V_{w_s} = Volume of water vapor at stack conditions, m^3

In terms of measured parameters the total gas sample volume is given by

$$V_{total} = V_m \frac{T_s}{T_m} \frac{\left(P_{bar} + \frac{\Delta H_{avg}}{13.6}\right)}{P_s} + K_w V_{l_c} \frac{T_s}{P_s} \qquad (23)$$

where:

V_m = Volume of gas sample through the dry gas meter (meter conditions), m^3

T_s = Average temperature of stack gas, °K

T_m = Average dry gas meter temperature, °K

P_{bar} = Barometric pressure at the orifice meter, mm Hg

ΔH_{avg} = Average pressure drop across the orifice meter, mm H_2O

13.6 = Specific gravity of mercury

P_s = Stack pressure, (Barometric ± static pressure), mm Hg

V_{1_c} = Total volume of liquid collected in impingers and silica gel, mℓ.

$$K_w = \frac{.00346 \text{ mm Hg} - m^3}{\text{m}\ell \ ^\circ K}$$

Variability in V_m is a combination of variability of the dry gas meter, inaccuracy of the calibration standard, and sampling train leaks, including those leaks undetected because of pump valve float. Assuming coefficients of variation of 1 percent, 0.4 percent, and 0.5 percent for the dry gas meter, calibration standard, and sampling train leaks, respectively, results in a $CV\{V_m\}$ = 1.2 percent.

$CV\{P_{bar}\}$ = 0.3 percent and $CV\{T_m\}$ = 0.5 percent are assumed from previous documents of this series (ref. 3).

Variability in ΔH_{avg} is believed to be primarily due to reading error (inclined manometer) and calibration error in determining ΔH@ (ΔH@ is the pressure drop across the orifice meter resulting in a .021 m^3/min (0.75 ft^3/min) flow rate at standard temperature and pressure). The pressure drop across the orifice, i.e., ΔH is relatively constant with no fluctuations and hence, easily read on an inclined monometer. Also, random reading error is averaged out because ΔH as used in equation (2) is an average of at least 12 readings. Therefore, the variability of the average pressure drop should be adequately characterized by a coefficient of variation of 1 percent, i.e.,$\{CV\ \Delta H_{avg}\}$ = 1 percent. These assumptions are summarized in table 3.

The estimated variance of V_{total}, as given by equation 3, section 2.5.3.3 can be derived from the following equation:

$$\begin{aligned}\sigma^2\left\{V_{total}\right\} = {} & (1.495)^2\sigma^2\left\{V_m\right\} + (.007)^2\sigma^2\left\{T_s\right\} + (-.009)^2\sigma^2\left\{T_m\right\} + \\ & (.003)^2\sigma^2\left\{P_{bar}\right\} + (.0002)^2\sigma^2\left\{\Delta H_{avg}\right\} + (-.005)^2\sigma^2\left\{P_s\right\} + \\ & (.002)^2\sigma^2\left\{V_{1_c}\right\} \\ = {} & .0039\end{aligned}$$

Table 3. Means and Variabilities of Parameters Affecting V_{total}

Parameter	Mean Value	Estimated CV for Repeatability
V_{1_c}	252 mℓ	$CV\{V_{1c}\} = 1.0\%$
T_s	405°K(730°R)	$CV\{T_s\} = 1.5\%$
P_s	707 mm(27.9 in. Hg)	$CV\{P_s\} = 0.9\%$
V_m	$1.7\ m^3 (60\ ft^3)$	$CV\{V_m\} = 1.2\%$
T_m	294°K(530°R)	$CV\{T_m\} = 0.5\%$
P_{bar}	760 mm Hg(29.9 in. Hg)	$CV\{P_{bar}\} = 0.3\%$
ΔH_{avg}	102 mm H_2O(4 in. H_2O)	$CV\{\Delta H\} = 1.0\%$

Since $CV^2\{V_{total}\} = \sigma^2\{V_{total}\}/(\bar{V}_{total})^2$, then $CV\{V_{total}\}$ is 2.9 percent. The most important variables in the determination of V_{total} are clearly T_s and V_m.

4.1.4 Beryllium Emission Rate, R.

The beryllium emission rate is calculated with equation 14 section 2.5.3.7. The variance of this determination can be calculated as follows (derived by using the Taylor series for the variance of a ratio):

$$CV^2\left\{R\right\} = CV^2\left\{W_t\right\} + CV^2\left\{Q_s\right\} + CV^2\left\{V_{total}\right\} \tag{24}$$

Using the coefficients of variation discussed in previous paragraphs $CV^2\{R\} = .14$ and $CV\{R\} = 37$ percent. This coefficient of variation is used here for illustrative purposes and is reasonable for sampling by experienced crews with adequate equipment.

4.2 ACTION OPTIONS

Suppose it has been determined as a result of the functional analysis and/or the reported data from the checking and auditing schemes, that the data quality is not consistent with suggested standards or with the user requirements. Poor data quality may result from (1) a lack of adherence to the control procedures given in section II--Operations Manual, or (2) the need for an improved method or instrumentation for taking the measurements. It is assumed in this section that (2) applies; that is, the data quality needs to be improved beyond that attainable by following the operational procedures given for the reference method.

The selection of possible actions for improving the data quality can best be made by those familiar with the measurement process and by the judicious use of the functional analysis as described in section 4.1. For each action, the variance analysis can be performed to estimate the variance, standard deviation, and coefficient of variation of the pertinent measurement(s). In some cases it is difficult to estimate the reduction in specific variances, which are required to estimate the precisions of the pertinent measurements. In such cases, an experimental study should be made of the more promising actions based on preliminary estimates of precision/bias and costs of implementation of each action. This preliminary analysis would follow the methods suggested below:

A0: Reference Method

A1: Crew Training (cost of $1000 per 20 stacks)

A2: Use of NBS Standard Reference Materials (cost of $750 per laboratory per year)

A3: Use of Programmable Minicalculator in lieu of Nomographs (cost of $350 per 20 stacks)

The costs given for each action are additional costs above that of the reference method. Assumptions concerning the reduction in variances (or improved precisions), are given for each action in the following paragraphs.

(1) Crew Training, A1. It is assumed that the coefficient of variation of R is reduced by 25 percent, or,

$$\mathrm{CV}\{R|A1\} = 0.75\ \mathrm{CV}\{R|A0\}.$$

The notation $CV\{R|A1\}$ denotes the "estimated CV of R given that A1 is implemented"; the vertical line is to be interpreted as "given that;" and the letters following the vertical line designate the implemented action as defined above. This results in a straightforward computation of $CV\{R|A1\} = 0.75 \times 25.7 = 19.3$ percent.

In estimating the cost for implementing this action it was assumed that one crew member be sent to a reputable source sampling school one week out of a year. The cost of the school plus subsistence is estimated to be $1000 in excess of salary and benefits. This cost is prorated over 20 source tests which is a reasonable number of tests per team per year.

(2) Standard Reference Materials, A2. The purchase and use of Standard Reference Materials (SRM's) acquired from the National Bureau of Standards is recommended for the analysis phase of the beryllium determination. From the SRM's, which are available in a set of three millipore filters in plastic cylinders certified at three different amounts of beryllium, a set of standard reference samples can be made for calibration of the spectrophotometer. Stated simply, the filters would be dissolved in an appropriate acid such as sulfuric acid or nitric acid and hydrogen peroxide and the resulting concentrated solutions stored in polyethelene containers. The working beryllium standard solutions are then diluted to the range of the NBS standard samples and the values compared and calibrated. These working standards are used throughout the measurement process to continually check the calibration of the spectrophotometer.

It is assumed that the coefficient of variation of R is reduced by 40 percent, or

$$CV\{R|A2\} = 0.60CV\{R|A0\}.$$

This results in a $CV\{R|A1\} = 15.4$ percent. An estimate of the cost based on the cost of a set of NBS filters, the additional manhours required to dilute and compare the standard working solutions, is $750 per laboratory per year. Assuming an average of 20 stacks sampled per year the figure is $750 per 20 source tests.

(3) Minicalculator in Lieu of Nomographs, A3. It is assumed that the coefficient of variation of V_n/V_s is reduced in accordance with the following equation:

$$CV\{R|A3\} = 0.90\ CV\ \{R|A0\}.$$

Therefore CV{R|A3} = 23.1 percent. An increase in precision and accuracy should be realized since exact values of C_p, ΔH@, and M_d would be used to determine isokinetic conditions rather than the mean values of their expected range as are now built into the nomograph. Also, routine calculation errors (i.e., error in setting and reading the nomograph) should be greatly reduced. A programmable mini-calculator can be purchased for about $350. Cost of programming should be more than recovered in sampling time saved as a result of using the calculator. The increased cost is taken as #350 per 20 source tests.

4.2.1 Actions Applied in Combination

In addition to treating each action separately, some of the actions can be combined to yield a further reduction in the variance of R or an increase in the precision. The assumptions stated above for the individual actions are applied in combination where appropriate to obtain an overall estimate. For example, applying actions A1 and A2 in combination results in a CV{R} = 11.58 percent. The costs are assumed to be additive, i.e. costs for applying A1 and A2 separately are added to obtain the cost of applying both A1 and A2. Similarly the costs are added for other combinations.

4.2.2 Comparison of Actions

The added cost per 20 stacks is plotted as a function of the precision of the estimated R's as measured by its coefficient of variation, CV{R}, in figure 12. Examination of the plotted results enables one to quickly identify the action or combination of actions which will yield results of some desired precision. In practice it may not be reasonable to insist that the data be of a specified precision, but that the cost

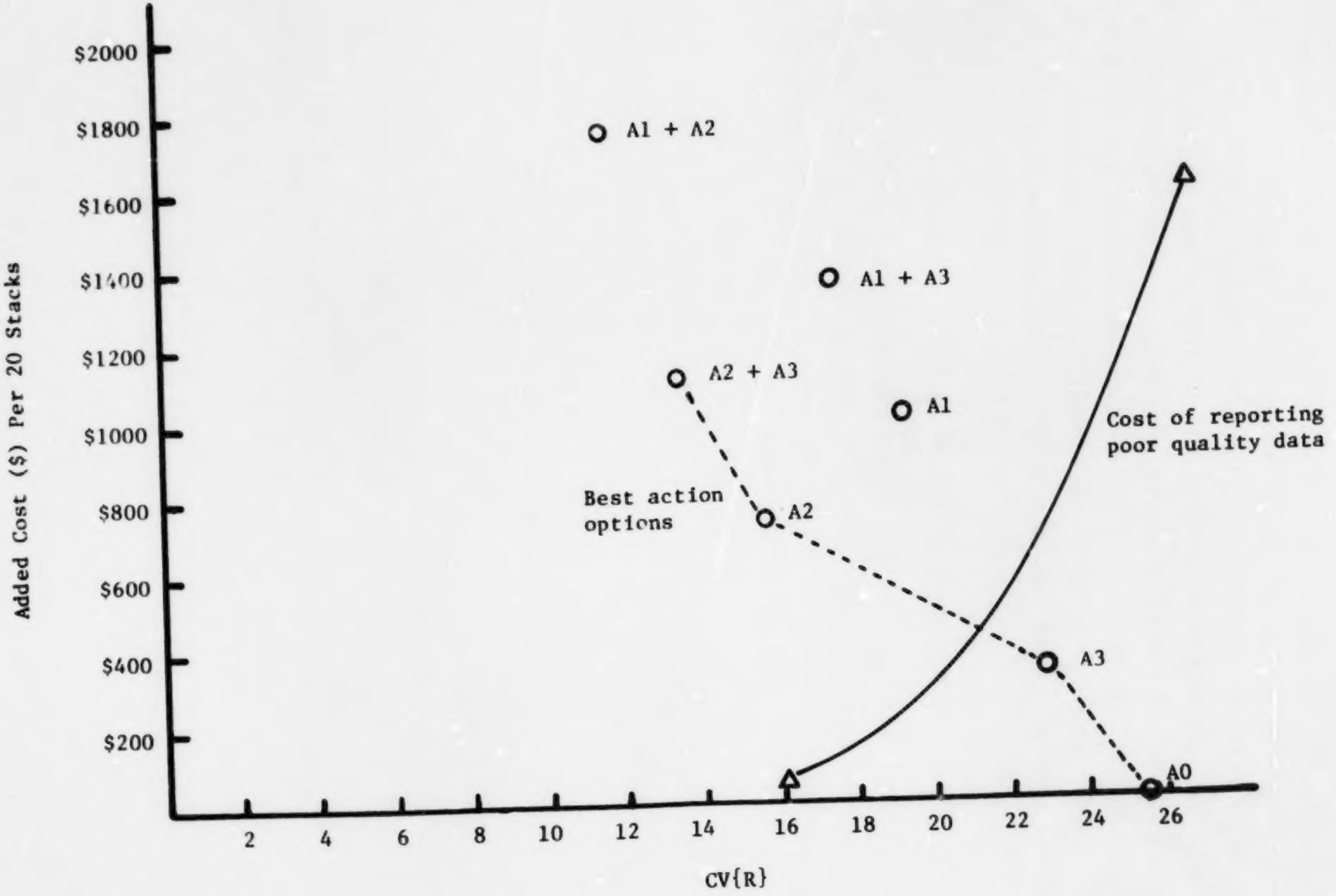


Figure 12. Added cost vs precision for selected action options for mercury emission rate determinations.

of reporting poor quality data is a rapidly increasing function of the data quality for data of variability exceeding a specified value. The selection of the best action option then becomes a trade-off between the overall cost and the expected precision to be achieved by its implementation. The cost of implementing an action plus the cost of reporting poor quality data are added to obtain an overall cost for the action. (An assumed function of the cost of reporting poor quality data is shown as the solid curve in figure 12 as an illustration only. Its exact shape and location on the graph would have to be determined from actual cost data).

4.3 PROCEDURES FOR PERFORMING A QUALITY AUDIT

"Quality audit" as used here implies a comprehensive system of planned and periodic audits to verify compliance with all aspects of the quality assurance program. Results from the quality audit provide an independent assessment of data quality. "Independent" means that the individuals performing, and as much as possible of the equipment used in the audit, are different from the regular field crew and equipment. From these data both bias and precision estimates can be made for the measurement process.

The auditor, i.e., the individual performing the audit, should have extensive background experience in source sampling, specifically with the characterization technique that he is auditing. He should be able to establish and maintain good rapport with field crews.

The functions of the auditor are summarized in the following list:

1. Observe procedures and techniques of the field team during on-site measurements.
2. Perform certain independent checks, and from previous training and experience, estimate the validity of the field crew's measurements.
3. Check/verify applicable records of equipment calibration checks and quality control charts in the field team's home laboratory.

4. Perform calculations using data obtained from the audit.
5. Compare the audit value with the field team's test value.
6. Inform the field team of the comparison results specifying any area(s) that need special attention or improvement.
7. File the records and forward the comparison results with appropriate comments to the manager.

4.3.1 Frequency of Audit

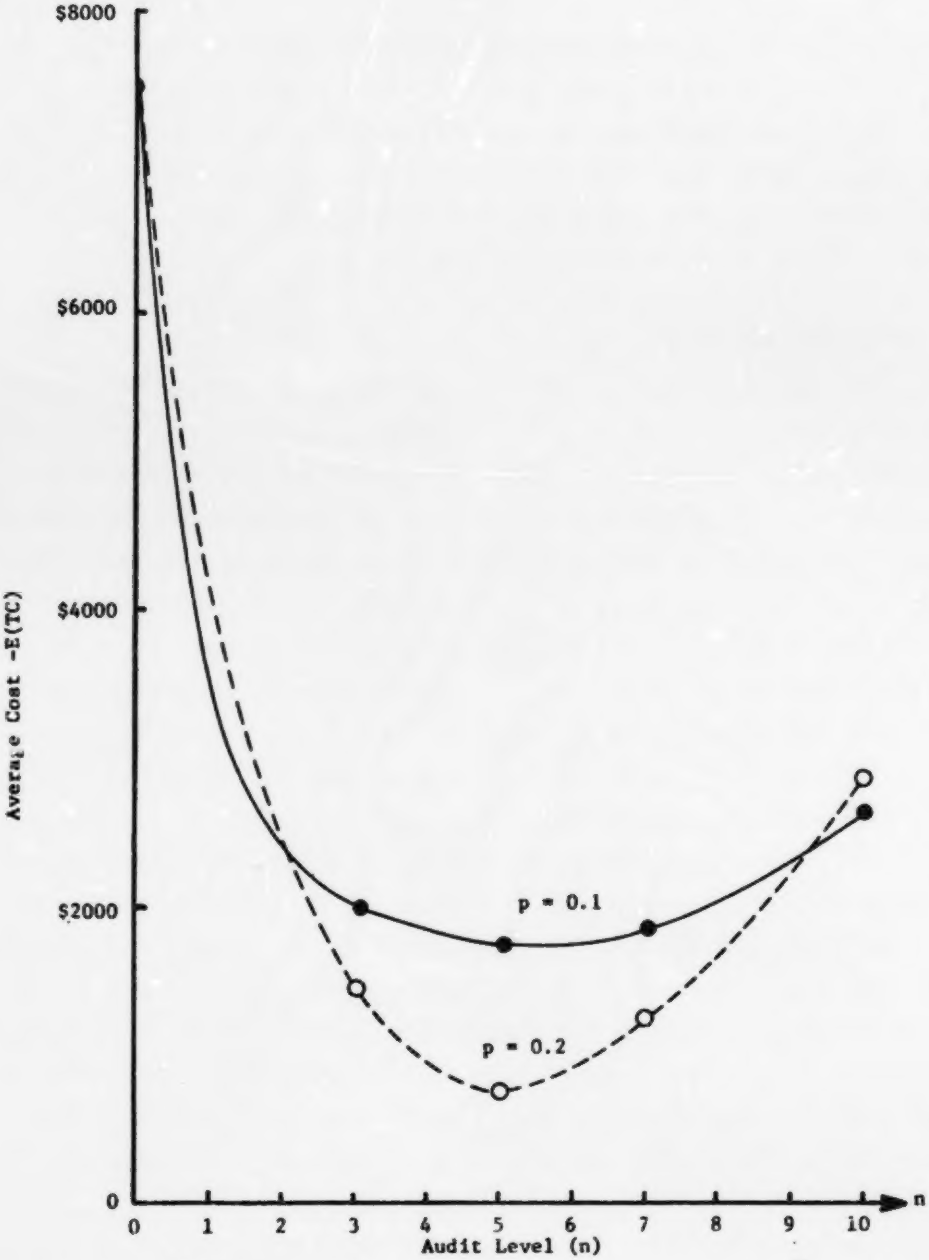
The optimum frequency of audit is a function of certain costs and desired level of confidence in the data quality assessment. A methodology for determining the optimum frequency using relevant costs is presented in subsection 4.4.4. For the costs assumed in this document, an optimum level of $n = 5$ for a lot size of $N = 20$ results as shown in figure 13. However, costs will vary among field teams and types of field tests. Therefore, the most cost effective auditing level will have to be derived using local cost data according to the procedure given in subsection 4.4.4 and in the final report on this contract.

4.3.2 Collecting Audit Information

While at the sampling site, the auditor should observe the field team's overall performance of the field test. Table 4 is a sample checklist of the operations to observe. Each item on the list should be checked yes or no according to whether it was performed as recommended in the operations manual and, if applicable, the result was within specified limits. Those items checked no should be explained under comments. No checklist can cover all situations; the auditor must utilize his good judgment and include other checks as deemed desirable for a specific situation.

A completed checklist with all yes checks implies that in the opinion of the auditor the measurement was made in such a manner that large biases resulting from poor technique are not likely to be present.

In addition to the above observations, the auditor should independently



p = Proportion defective measurements in the "lot"
P{Acc. lot with p} ≤ 0.1

Figure 13. Average cost vs audit level (n).

Table 4. Beryllium Emission Determination Checklist to be Used by Auditor

YES	NO	OPERATION
		EQUIPMENT PREPARATION AND CHECK
___	___	1. Sampling train assembled and leak checked.
___	___	2. Probe and filter box heaters checked and set for proper temparatures.
___	___	3. Stack gas temperature measuring system assembled and checked for proper operation by comparing to a mercury in glass thermometer.
___	___	4. Stack gas velocity measuring system assembled and checked for proper operation.
___	___	5. Orsat analyzer assembled and checked (if used).
		PRELIMINARY MEASUREMENTS
___	___	6. Selection of traverse points according to Method 1.
___	___	7. Moisture content by Method 4, or equivalent.
___	___	8. Molecular weight by Method 3, or equivalent.
___	___	9. Measurement of stack dimensions.
___	___	10. Mark probe for sampling at traverse points.
		SAMPLE COLLECTION
___	___	11. Equal sampling time at each traverse point.
___	___	12. Probe temperature satisfactory throughout the test.
___	___	13. Filter box temperature at appropriate temperature to prevent condensation throughout the test.
___	___	14. Sample gas temperature at last impinger ≈ 20°C (68°F) throughout the test.
___	___	15. Isokinetic sampling checked and adjusted if necessary at least every 5 minutes.
___	___	16. Leak check of sampling train before and after each run.
		SAMPLE RECOVERY
___	___	17. Satisfactory handling and movement of probe and filter to sample recovery area.
___	___	18. Recovery area satisfactory (i.e., space, cleanliness, etc.)

Table 4. Beryllium Emission Determination Checklist to be Used by Auditor (continued)

YES	NO	OPERATION
___	___	19. Sample recovery procedure adequate.
___	___	20. Proper labeling of sample containers.
___	___	21. Determination of moisture content procedure adequate.
		ANALYSIS
___	___	22. Proper calibration of the spectrophotometer.
___	___	23. Careful dilution techniques used in diluting field samples and standard samples.
		DOCUMENTATION
___	___	24. All information recorded on data sheet as obtained.
___	___	25. All unusual conditions recorded.
		COMMENTS

determine the stack dimensions. This should be carried out with the realization that the measurement is to be used as an estimate of the average dimension of the stack. Therefore, for example, a stack which could be out of round should have its diameter measured from as many sampling ports as possible and the average diameter used as the stack diameter in subsequent calculations. Record the cross-sectional area, A_{sa}, on the form of figure 14.

The auditor should obtain from the field team a complete set of test data, i.e., the data form of figure 5 filled in through the section on recorded test data, for all sample runs.

In the field team's home laboratory the auditor should verify, by checking calibration records and field data sheets from previous field tests, that the performance criteria as given in table 1 of section III have been satisfied over the period since the last audit. Also, using his own calibrated standards, perform the following calibration checks.

1. Using his own calibrated wet test meter, or equivalent, calibrate the dry gas meter and the orifice meter as directed in subsection 2.2.3 of section II. Record these audit values as γ_a and $\Delta H@$ in the form in figure 14.

2. Determine the sampling nozzle diameter, D_{na}, according to the procedure of subsection 2.2.1. Calculate the cross-sectional area, A_{na}, in square meters by

$$A_{na} = 0.25 \times \frac{22}{7} \times (D_{na})^2 \times \frac{1}{10^6} = \underline{\qquad\qquad} \frac{m^2}{mm^2} \qquad (25)$$

$$A_{na} = 7.85 \times 10^{-7} \times (D_{na})^2 = \underline{\qquad\qquad} m^2.$$

Record D_{na} and A_{na} on the audit data sheet of figure 14.

3. Using a calibrated pitot tube, calibrate the field pitot tube according to the procedure given in subsection 2.1.2, of the quality assurance document of this series for Method 2. The field pitot tube

AUDIT MEASUREMENTS

STACK CROSS-SECTIONAL AREA, A_{s_a} __________ m^2
DRY GAS METER CALIBRATION, Y_a __________ Dimensionless
ORIFICE METER CALIBRATION, $\Delta H@_a$ __________ mm H_2O
SAMPLING NOZZLE DIAMETER, D_{na} __________ mm
SAMPLING NOZZLE CROSS-SECTIONAL AREA, A_n __________ m^2
PITOT TUBE CALIBRATION COEFFICIENT, C_{p_a} __________ Dimensionless

CALCULATIONS

1. $V_{ma} = V_m \times Y =$ ________ m^3

2. $V_{ma_s} = V_m \frac{T_s}{T_m} \frac{\left(P_{bar} + \frac{\Delta H_{avg}}{13.6}\right)}{P_s} =$ ______ m^3 at stack conditions

3. $V_{wa_s} = .00346\ V_{1_c} \frac{T_s}{P_s} =$ _______ m^3

4. $V_{total_a} = V_{m_s} + V_{w_s} =$ ________ m^3

5. $V_{ma_{std}} = .3874\ V_m \frac{\left(P_{bar} + \frac{\Delta H_{avg}}{13.6}\right)}{} =$ _______ m^3

6. $B_{wo_a} = \frac{0.0013\ V_{1_c}}{V_{m_{std}} + 0.0013} =$ _________ dimensionless

7. $M_{sa} = M_d(1-B_{wo}) + 18B_{wo} =$ _________ g/g-mole

8. $(v_{sa})_{avg} = 34.96\ C_p\ (\sqrt{\Delta P})_{avg} \sqrt{\frac{(T_s)_{avg}}{P_s\ M_s}} =$ ________ m/sec

9. $W_{ta} = V_f C_f - V_w C_w - V_a C_a =$ _________ ug

10. $R_a = \frac{.0864\ W_t (v_s)_{avg}\ A_s}{V_{total}} =$ _____ g/day

11. $I_a = \frac{100\ V_{total}}{A_n\ \theta (v_s)_{avg}} =$ _______ percent

12. $\bar{d} = \frac{R-R_a}{R_a} \times 100 =$ _________ percent

Figure 14. Sample form for recording audit data

is calibrated in the sampling configuration, i.e., the pitot tube is strapped to the sampling probe and isokinetic sampling conditions are maintained when reading the velocity pressure head with the field pitot tube. The average coefficient, C_{p_a}, applicable to the ΔP range measured in the field test is determined from this calibration data. Record C_{p_a} on the form in figure 14.

4.3.3 Treatment of Audit Data

Using the above audit data where applicable and the raw field data, perform the calculations indicated in figure 14. All variables are in the same units as used in subsection 2.5.3 of the operations manual. In figure 14, audit measurements and/or parameters computed using audit measurements are subscripted with an a. Parameters such as $(\sqrt{\Delta P})_{avg}$, $(T_s)_{avg}$, M_s, and B_{wo} should be calculated by the auditor from the original field data. All calculations are recorded on the audit data sheet of figure 14.

The auditor's report of a specific field test to the manager should include copies of (1) a completed data sheet from the field team (fig. 5, pp. 41-3), (2) a completed checklist with comments (table 4, p. 110), (3) a completed audit data sheet with calculations (fig. 14, p. 109), and (4) a summary of the field team's strong/weak points with an overall numerical rating and recommended actions as discussed in the following subsection.

4.3.4 Overall Evaluation of Field Team Performance

In a summary-type statement, the field team should be evaluated on its overall performance. Using the checklist filled out in the field (table 4) in conjunction with the results of the comparison of audit and field team values of R and the circumstances under which the test was performed, **field team** perforamnce could be rated on a scale of 1 to 5 as follows:

5 - Excellent
4 - Above average
3 - Average

2 - Acceptable, but below average
1 - Unacceptable

Justification for the rating in the form of a list of the team's strong/weak points with recommendations for improving weak points should be included in the auditor's report.

4.4 DATA QUALITY ASSESSMENT

Two aspects of data quality assessment are considered in this section. The first considers a means of estimating the precision and accuracy of the reported data, e.g., reporting the bias, if any, and the standard deviation associated with the measurements. The second consideration is that of testing the data quality against given standards using sampling by variables. For example, lower and upper limits, L and U, may be selected to include a large percentage of the measurements and outside of which it is desired to control the percentage of measurements to, say, less than 10 percent. If the data quality is not consistent with these limits, L and U, then action is taken to correct the possible deficiency before future field tests are performed and to correct the previous data when possible.

The determination of the audit level is indicated by using estimated costs associated with falsely inferring that good (poor) quality data are of poor (good) quality and with auditing n stacks. In addition, prior information concerning data quality is assumed in order to determine an expected or average cost resulting from the statistical sampling plan. The cost estimates provided herein are assumed for the purpose of illustrating the methodology. It is emphasized that managers need to supply their own costs in making such analyses.

4.4.1 Estimating the Precision/Accuracy of the Reported Data

Methods for estimating the precision (standard deviation) and bias of the mercury emission rate ($\overline{R}$) measurements were given in section 4.1. This section will indicate how the audit data collected in accordance with the procedure described in section 4.3 will be utilized to estimate the precision and accuracy of the measure of interest. Similar techniques can also be used by a specific firm or team to assess their own measurements. However, in this case no bias data among firms can be obtained. The audit data collected as a result of following the procedures in the previous section are the measured and audited values of $\overline{R}$ and the difference.

$$d_j = \frac{\overline{R}_j - \overline{R}_{aj}}{\overline{R}_{aj}} \times 100 \qquad (26)$$

where $\overline{R}_j$ = Field measured value of the beryllium emission rate, average of three replicates, and

$\overline{R}_{aj}$ = Audited value of the beryllium emission rate, average of three replicates.

Let the mean and standard deviation of the differences d_j, $j = 1, \ldots n$ field tests be denoted by $\overline{d}$ and s_d, respectively. Thus,

$$\overline{d} = \sum_{j=1}^{n} d_j/n, \qquad (27)$$

$$s_d = \left[\sum_{j=1}^{n} (d_j - \overline{d})^2/(n-1)\right]^{1/2}. \qquad (28)$$

4.4.2 Statistical Tests

The mean $\overline{d}$ is an estimate of the relative bias in the measurements (i.e., relative to the audited value). Assuming the audited data to be unbiased, the existence of a bias in the field data can be checked by the appropriate t-test, i.e.,

$$t_{n-1} = \frac{\overline{d} - 0}{s_d/\sqrt{n}} . \tag{29}$$

See reference 23 for a discussion of the t-test. If t is significantly large in absolute values, i.e., greater than the tabulated value of t with n - 1 degrees of freedom, which is exceeded by chance only 5 percent of the time, then the bias is considered to be real and some check should be made for a possible cause of the bias. If t is not significantly large, then the bias should be considered zero or negligible.

The standard deviation, s_d, is a function of both the standard deviation of the field measurements and of the audit measurements. Assuming the audited measurements are obtained with much greater precision than the field measurements, then the calculated s_d is an estimate of the standard deviation of the field measurement. Furthermore, since s_d is in percent, it is an estimate of the coefficient of variation, CV{R}. Table 5, contains an example calculation of $\overline{d}$ and s_d, starting with the differences for a sample size of n = 5. See the final report on the contract for further information concerning this result.

The calculated standard deviation can then be utilized to check the reasonableness of the assumption made in section 4.1 concerning CV{R} = 25.7 percent for R = 2.0 g/d, for example. (Remember that CV{R_c} is equal to σ{d}.) The calculated standard deviation, s_d, may be directly checked against the assumed value, σ{d}, by using the statistical test procedure

$$\frac{\chi^2}{f} = \frac{s_d^2}{\sigma^2\{d\}}, \tag{30}$$

where χ^2/f is the value of a random variable having the chi-square distribution with f = n - 1 degrees of freedom. If χ^2/f is larger than the tabulated value exceeded only 5 percent of the time, then it would be concluded that the test procedure is yielding results with more variability than is acceptable due to some assignable cause of large variation.

Table 5. Computation of the Mean Difference, $\bar{d}$, and Standard Deviation of the Differences, s_d.

General formulas		Specific example	
1. $d_j = \frac{\bar{R}_j - \bar{R}_{aj}}{\bar{R}_{aj}} \times 100$		Data	
d_1	d_1^2	32.0	1024.0
d_2	d_2^2	19.5	380.3
d_3	d_3^2	10.3	106.1
d_4	d_4^2	12.1	146.4
d_5	d_5^2	14.6	213.2
$\sum d_j$	$\sum d_j^2$	88.5	1870.0
2. $\bar{d} = \frac{\sum d_j}{n}$		$\bar{d} = 17.7\%$	
3. $s_d^2 = \frac{\sum d_j^2 - (\sum d_j)^2 / n}{(n-1)}$		3. $s_d^2 = 75.9$	
4. $s_d = \sqrt{s_d^2}$		4. $s_d = 8.7\%$	

The measured values should be reported along with the estimated bias, $\bar{d}$, standard deviation, s_d, the number of audits, n, and the total number of field tests, N, sample $(n \leq N)$. Estimates, i.e., s_d and $\bar{d}$, which are significantly different from the assumed population parameters should be identified on the data sheet. For example, based on the data of table 5, if the field team reported a value of $\bar{R} = 2.0$ g/d for one of the N field tests not audited, then that measurement would be reported as

1. Measured value, $\bar{R} = 2.0$ g/d
2. Calculated bias, $\hat{\tau}\{R\} = \bar{d} \times \bar{R} = .35$ g/d
3. Calculated standard deviation, $\hat{\sigma}\{R\} = s_d \times \bar{R} = 0.174$ g/d
4. Auditing level, $n = 5$, $N = 20$

From the above data, users of the data can calculate confidence limits appropriate to what the data are to be used for.

The t-test and χ^2-test described above, and in further detail in the final report on this contract, are used to check on the biases and standard deviations separately. In order to check on the overall data quality as measured by the percent of measurement deviations outside prescribed limits, it is necessary to use the approach described in subsection 4.4.3 below.

4.4.3 Sampling by Variables

Because the lot size (i.e., the number of field tests performed by a team or laboratory during a particular period, normally a calendar quarter) is small, $N = 20$, and consequently, the sample size is small on the order of $n = 3$ to 7, it is important to consider a sampling by variables approach to assess the data quality with respect to prescribed limits. That is, it is desired to make as much use of the data as possible. In the variables approach, the means and standard deviations, of the sample of n audits are used in making a decision concerning the data quality.

Some background concerning the assumptions and the methodology is repeated below for convenience. However, one is referred to one of a number of publications having information on sampling by variables; e.g., see references 24-27. The discussion below will be given in regard to the specific problem herein which has some unique features as compared with the usual variable sampling plans.

In the following discussion, it is assumed that only $\overline{R}$ is audited as directed in sections 4.3.2.1 and 4.3.2.2.

The difference between the team-measured and audited value of $\overline{R}$ is designated as d_j, and the mean difference over n audits by $\bar{d}$, that is,

$$\bar{d} = \frac{100}{n} \sum_{j=1}^{n} \left(\frac{\overline{R}_j - \overline{R}_{aj}}{\overline{R}_{aj}} \right). \tag{31}$$

Theoretically, $\overline{R}_j$ and $\overline{R}_{aj}$ should be measures of the same beryllium emission rate, and their difference should have a mean of zero on the average. In addition, their differences should have a standard deviation approximately equal to that associated with measurements of $\overline{R}_j$ separately.

Assuming three standard deviation limits (using the assumed CV = 25.7 percent as derived in the variance analysis of subsection 4.1), the values -3(25.7%) = -77.1 percent and 3(25.7%) = 77.1 percent define lower and upper limits, L and U, respectively, outside of which it is desired to control the proportion of differences, d_j. Following the method given in reference 24, a procedure for applying the variables sampling plan is described below. Figures 15 and 16 illustrate examples of satisfactory and unsatisfactory data quality with respect to the prescribed limits L and U.

The variables sampling plan requires the sample mean difference, $\bar{d}$; the standard deviation of these differences, s_d; and a constant, k, which is determined by the value of p, the proportion of the differences outside the limits of L and U. For example, if it is desired to control at 0.10 the probability of not detecting lots with data quality p equal

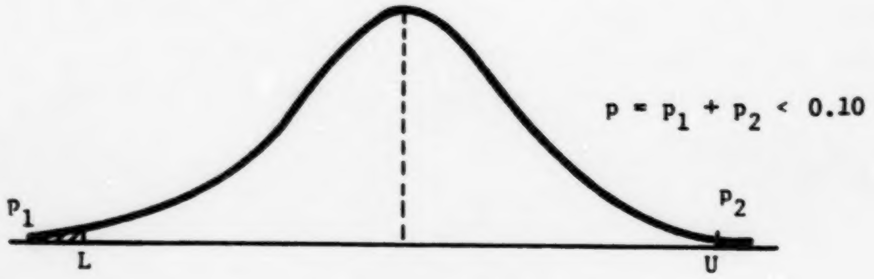

$p = p_1 + p_2 < 0.10$
p_1
p_2
L
U

Figure 15. Example illustrating $p < 0.10$ and satisfactory data quality.

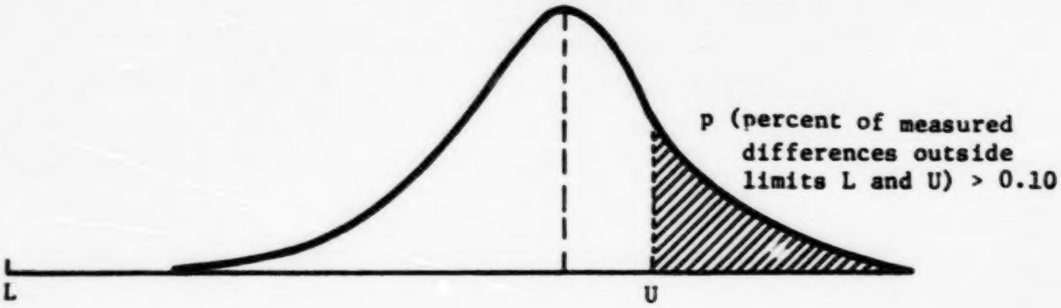

p (percent of measured differences outside limits L and U) > 0.10
L
U

Figure 16. Example illustrating $p > 0.10$ and unsatisfactory data quality.

to 0.10 (or 10% of the individual differences outside L and U) and if the sample size n = 5, then the value of k can be obtained from a table such as table 6. The values of $\bar{d}$ and s_d are computed in the usual manner; see table 5 for formulas and a specific example. Given the above information, the test procedure is applied and subsequent action is taken in accordance with the following criteria:

1. If both of the following conditions are satisfied:

$$\bar{d} - k\, s_d \geq L = -77.1 \text{ percent},$$

$$d + k\, s_d \leq U = +77.1 \text{ percent}$$

the individual differences are considered to be consistent with the prescribed data quality limits and no corrective action is required.

2. If one or both of these inequalities is violated, possible deficiencies exist in the measurement process as carried out for that particular lot (group) of field tests. These deficiencies should be identified and corrected before future field tests are performed. Data corrections should be made when possible, i.e., if a quantitative basis is determined for correction.

Table 6. Sample Plan Constants, k for P{Not Detecting a Lot with Proportion P Outside Limits L and U} $\leq$ 0.1

Sample size n	k(p = 0.2)	k(p = 0.1)
3	3.039	4.258
5	1.976	2.742
7	1.721	2.334
10	1.595	2.112
12	1.550	2.045

Using the values of $\bar{d}$ and s_d in table 6, $k = 2.742$ for a sample size $n = 5$, and $p = 0.10$, the test criteria can be checked; i.e.,

$$\bar{d} - k\, s_d = 17.7 - 2.742(8.7) = -6.2 > L = -77.1 \text{ percent}$$

and

$$\bar{d} + k\, s_d = 17.7 + 2.742(8.7) = 41.6 < U = 77.1 \text{ percent.}$$

Therefore, both conditions are satisfied and the lot of $N = 20$ measurements is consistent with the prescribed quality limits. The plan is designed to protect against not detecting lots with 10 percent or more defects (deviations falling outside the designated limits L and U) with a risk of 0.10; that is, on the average, 90 percent of the lots with 10 percent or more defects will be detected by this sampling plan.

4.4.4 Cost Versus Audit Level

The determination of the audit level (sample size n) to be used in assessing the data quality with reference to prescribed limits L and U can be made on a statistical basis by defining acceptable risks for type I and type II errors, knowing or estimating the quality of the incoming data, and specifying the described level of confidence in the reported data, or on a cost basis as described herein. In this section, cost data associated with the audit procedure are estimated or assumed for the purpose of illustrating a method of approach and identifying which costs should be considered.

A model of the audit process, associated costs, and assumptions made in the determination of the audit level is provided in figure 17. It is assumed that a collection of source emissions tests for N stacks is to be made by a particular firm, and that n measurements ($n \leq N$) are to be audited at a cost, $C_A = b + cn$, where b is a constant independent for n, and c is the cost per stack measurement audited. In order to make a specific determination of n, it is also necessary to make some assumptions about the

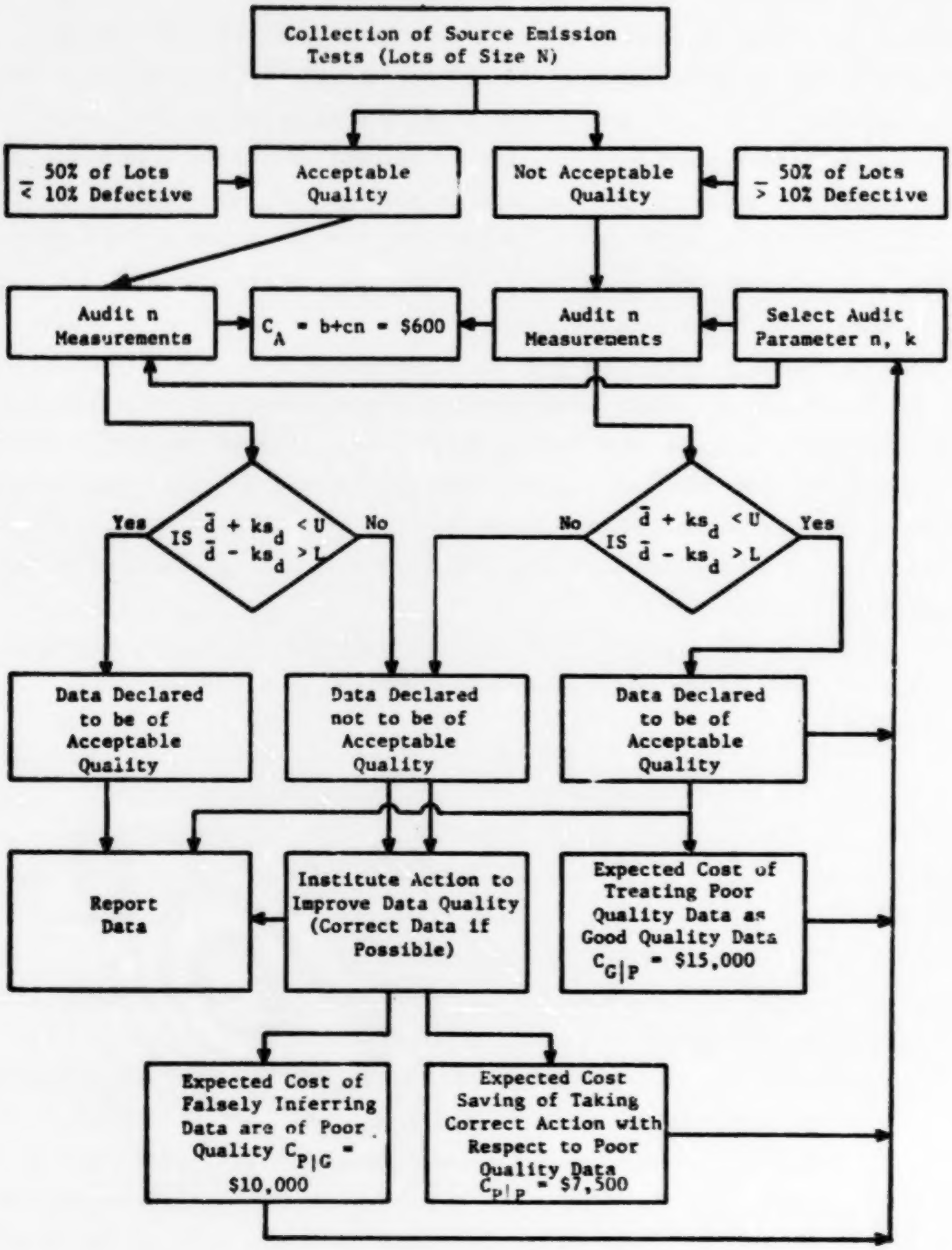

Collection of Source Emission Tests (Lots of Size N)
≤ 50% of Lots 10% Defective
Acceptable Quality
Not Acceptable Quality
≥ 50% of Lots 10% Defective
Audit n Measurements
C_A = b+cn = $600
Audit n Measurements
Select Audit Parameter n, k
Yes
IS $\bar{d} + ks_d < U$, $\bar{d} - ks_d > L$
No
No
IS $\bar{d} + ks_d < U$, $\bar{d} - ks_d > L$
Yes
Data Declared to be of Acceptable Quality
Data Declared not to be of Acceptable Quality
Data Declared to be of Acceptable Quality
Report Data
Institute Action to Improve Data Quality (Correct Data if Possible)
Expected Cost of Treating Poor Quality Data as Good Quality Data $C_{G|P}$ = $15,000
Expected Cost of Falsely Inferring Data are of Poor Quality $C_{P|G}$ = $10,000
Expected Cost Saving of Taking Correct Action with Respect to Poor Quality Data $C_{P|P}$ = $7,500

Figure 17. Flow chart of the audit level selection process.

quality of the source emissions data from several firms. For example, it is assumed in this analysis that 50 percent of the data lots are of good quality, i.e., one half of the firms are adhering to good data quality assurance practice, and that 50 percent of the data lots are of poor quality. Based on the analysis in section 4.1, good quality data is defined as that which is consistent with the estimated precision/bias using the reference method. Thus, if the data quality limits L and U are taken to be the lower and upper 3σ limits corresponding to limits used in a control chart, the quality of data provided by a firm adhering to the recommended quality assurance procedures should at most contain about 0.3 percent defective measurements (i.e., outside the limits defined by L and U). Herein, good quality data are defined as that containing at most 10 percent defective measurements. The definition of poor quality data is somewhat arbitrary, for this illustration it is taken as 25 percent outside L and U.

In this audit procedure the data are declared to be of acceptable quality if both of the following inequalities are satisfied

$$\bar{d} + ks_d < U \tag{32}$$

$$\bar{d} - ks_d > L, \tag{33}$$

where $\bar{d}$ and s_d are the mean and standard deviation of the data quality characteristic (i.e., the difference of the field and audited measurements) being checked, and not of desired quality if one or both inequalities is violated, as described in section 4.3. The cost associated with these actions are assumed to be as follows:

C_A = Audit cost = b + cn. b is assumed to be zero for this example and c is taken as \$600/measurement.

$C_{P|G}$ = Cost of falsely inferring that the data are of poor quality, P, given that the data are of good quality, G. This cost is assumed to be one-half the cost of collecting emissions data for N = 20 stacks (i.e., 0.5 × \$1000 × 20 = \$10,000). This cost would include that of searching for an assignable cause of the inferred data deficiency when none exists, partial repetition of data collection, decision resulting in the purchase of equipment to reduce emission levels of specific pollutants, etc.

$C_{P|P}$ = Cost savings resulting from correct identification of poor quality data. This cost is taken to be \$7,500, i.e., equal to one-half of $C_{P|G}$ or equal to 0.375 × \$1,000 × 20, the total cost of data collection.

These costs are given in figure 17. These cost data are then used in conjunction with the a priori information concerning the data quality to select an audit level n. Actually, the audit procedure requires the selection of the limits L and U, n, and k. L and U are determined on the basis of the analysis of section 4.1. The value of k is taken to be the value associated with n in table 6 of section 4.4.3, i.e., the value selected on a statistical basis to control the percentage of data outside the limits L and U. Thus, it is only necessary to vary n and determine the corresponding expected total cost E(TC) using the following cost model.

$$E(TC) = -C_A - 0.5\, P_{P|G}\, C_{P|G} + 0.5\, P_{P|P}\, C_{P|P} - 0.5\, P_{G|P}\, C_{G|P} \qquad (34)$$

where the costs are as previously defined. The probabilities are defined in a similar way to the corresponding costs.

$P_{P|G}$ = Probability that a lot of good quality data is falsely inferred to be of poor quality due to the random variations in the sample mean $\bar{d}$ and standard deviation, s_d, in small samples of size n.

$P_{P|P}$ = Probability that a lot of poor quality data is correctly identified as being of poor quality.

$P_{G|P}$ = Probability that a lot of poor quality data is incorrectly judged to be of good quality due to sampling variations of $\bar{d}$ and s.

These three probabilities are conditional on the presumed lot quality and are preceded by a factor of 0.5 in the total cost model to correspond to the assumed percentage of good (poor) quality data lots.

In order to complete the determination of n, it is necessary to calculate each of the conditional probabilities using the assumptions stated for a series of values of n (and associated k which is given in table 11). The computational procedure is given in the Final Report of this contract. These calculations were made for the cases $n = 3, 5, 7$, and 10 and for two degrees of control on the quality of the data that can be tolerated, i.e., $p = 0.2$ and $p = 0.1$, the portion outside the limits L and U for which it is desired to accept the data as of good quality with probability less than or equal to 0.10. These computed probabilities are then used in conjunction with the costs associated with each condition, applying equation (34) to obtain the average cost versus sample size n for the two cases $p = 0.1$ and 0.2. The curves obtained from these results are given in figure 13. It can be seen from these curves that the minimum cost is obtained by using $n \simeq 5$ independent of p. However, it must be recognized that the costs used in the example are for illustrative purposes and may vary from one region to another, thus within the reasonable uncertainty of the estimated costs, values of n between 3 and 7 would seem to be reasonable. The assumed costs suggest that $p = 0.2$ is more cost effective, which tends to permit data of poorer quality to be accepted.

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APPENDIX A

sample is digested in an acid solution and analyzed by atomic absorption spectrophotometry.

1.2 *Applicability.*—This method is applicable for the determination of beryllium emissions in ducts or stacks at stationary sources. Unless otherwise specified, this method is not intended to apply to gas streams other than those emitted directly to the atmosphere without further processing.

2. *Apparatus*—2.1 *Sampling train.*—A schematic of the sampling train used by EPA is shown in figure 104–1. Commercial models of this train are available, although construction details are described in APTD-0581,[1] and operating and maintenance procedures are described in APTD-0576. The components essential to this sampling train are the following:

2.1.1 *Nozzle.*—Stainless steel or glass with sharp, tapered leading edge.

2.1.2 *Probe.*—Sheathed Pyrex[2] glass. A heating system capable of maintaining a minimum gas temperature in the range of the stack temperature at the probe outlet during sampling may be used to prevent condensation from occurring.

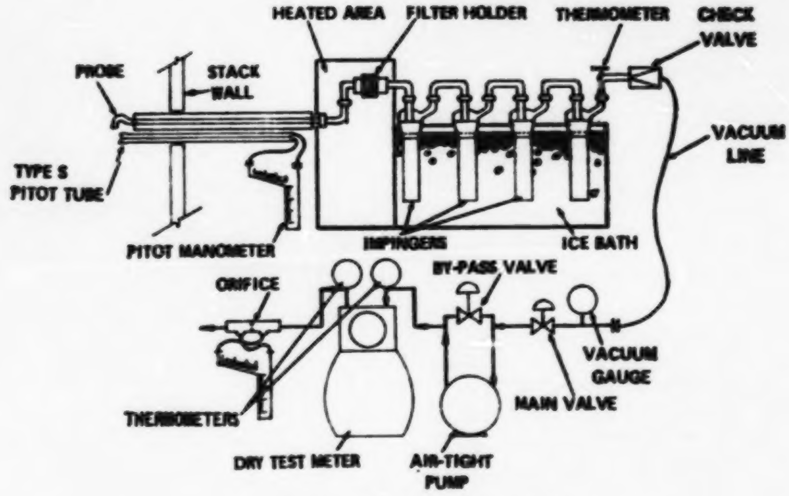

Figure 104-1. Beryllium sampling train

2.1.3 *Pitot tube.*—Type S (figure 104–2), or equivalent, with a coefficient within 5 percent over the working range, attached to probe to monitor stack gas velocity.

2.1.4 *Filter holder.*—Pyrex glass. The filter holder must provide a positive seal against leakage from outside or around the filter. A heating system capable of maintaining the filter at a minimum temperature in the range of the stack temperature may be used to prevent condensation from occurring.

2.1.5 *Impingers.*—Four Greenburg-Smith impingers connected in series with glass ball joint fittings. The first, third, and fourth impingers may be modified by replacing the tip with a ½-inch i.d. glass tube extending to one-half inch from the bottom of the flask.

2.1.6 *Metering system.*—Vacuum gauge, leakless pump, thermometers capable of measuring temperature to within 5° F, dry gas meter with 2 percent accuracy, and related equipment, described in APTD-0581, to maintain an isokinetic sampling rate and to determine sample volume.

2.1.7 *Barometer.*—To measure atmospheric pressure to ± 0.1 in Hg.

2.2 *Measurement of stack conditions (stack pressure, temperature, moisture and velocity)*—2.2.1 *Pitot tube.*—Type S, or equivalent, with a coefficient within 5 percent over the working range.

2.2.2 *Differential pressure gauge.*—Inclined manometer, or equivalent, to measure velocity head to within 10 percent of the minimum value.

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[1] These documents are available for a nominal cost from the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Va. 22151.

[2] Mention of trade names on specific products does not constitute endorsement by the Environmental Protection Agency.

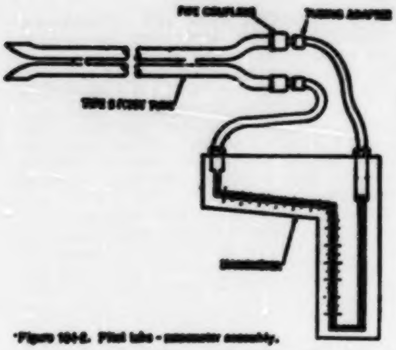

*Figure 104-2. Pitot tube - manometer assembly.

2.2.3 *Temperature gage.*—Any temperature measuring device to measure stack temperature to within 5° F.

2.2.4 *Pressure gage.*—Pilot tube and inclined manometer, or equivalent, to measure stack pressure to within 0.1 in Hg.

2.2.5 *Moisture determination.*—Wet and dry bulb thermometers, drying tubes, condensers, or equivalent, to determine stack gas moisture content to within 1 percent.

2.3 *Sample recovery*—2.3.1 *Probe cleaning rod.*—At least as long as probe.

2.3.2 *Leakless glass sample bottles.*—500 ml.

2.3.3 *Graduated cylinder.*—250 ml.

2.3.4 *Plastic jar.*—Approximately 300 ml.

2.4 *Analysis*—2.4.1 *Atomic absorption spectrophotometer.*—To measure absorbance at 234.8 nm. Perkin Elmer Model 303, or equivalent, with N_2O/acetylene burner.

2.4.2 *Hot plate.*

2.4.3 *Perchloric acid fume hood.*

3. *Reagents*—3.1 *Stock reagents.*—3.1.1 *Hydrochloric acid.*—Concentrated.

3.1.2 *Perchloric acid.*—Concentrated, 70 percent.

3.1.3 *Nitric acid.*—Concentrated.

3.1.4 *Sulfuric acid.*—Concentrated.

3.1.5 *Distilled and deionized water.*

3.1.6 *Beryllium powder.*—98 percent minimum purity.

3.2 *Sampling*—3.2.1 *Filter.* — Millipore AA, or equivalent. It is suggested that a Whatman 41 filter be placed immediately against the back side of the Millipore filter as a guard against breaking the Millipore filter. In the analysis of the filter, the Whatman 41 filter should be included with the Millipore filter.

3.2.2 *Silica gel.*—Indicating type, 6 to 16 mesh, dried at 350° F for 2 hours.

3.2.3 *Distilled and deionized water.*

3.3 *Sample recovery*—3.3.1 *Distilled and deionized water.*

3.3.2 *Acetone.*—Reagent grade.

3.3.3 *Wash acid.*—1:1 V/V hydrochloric acid-water.

3.4 *Analysis.*—3.4.1 *Sulfuric acid solution, 12 N.*—Dilute 333 ml of concentrated sulfuric acid to 1 l with distilled water.

3.4.2 *25 percent V/V hydrochloric acid-water.*

3.5 *Standard beryllium solution*—3.5.1 *stock solution.*—1 μg/ml beryllium. Dissolve 10 mg of beryllium in 80 ml of 12 N sulfuric acid solution and dilute to a volume of 1000 ml with distilled water. Dilute a 10 ml aliquot to 100 ml with 25 percent V/V hydrochloric acid, giving a concentration of 1 μg/ml. This dilute stock solution should be prepared fresh daily. Equivalent strength (in beryllium) stock solutions may be prepared from beryllium salts as $BeCl_2$ and $Be(NO_3)_2$ (98 percent minimum purity).

4. *Procedure.* 4.1 Guidelines for source testing are detailed in the following sections. These guidelines are generally applicable; however, most sample sites differ to some degree and temporary alterations such as stack extensions or expansions often are required to insure the best possible sample site. Further, since beryllium is hazardous, care should be taken to minimize exposure. Finally, since the total quantity of beryllium to be collected is quite small, the test must be carefully conducted to prevent contamination or loss of sample.

4.2 Selection of a sampling site and minimum number of traverse points.

4.2.1 Select a suitable sampling site that is as close as practicable to the point of atmospheric emission. If possible, stacks smaller than 1 foot in diameter should not be sampled.

4.2.2 The sampling site should be at least 8 stack or duct diameters downstream and 2 diameters upstream from any flow disturbance such as a bend, expansion or contraction. For a rectangular cross-section, determine an equivalent diameter from the following equation:

$$D_e=\frac{2LW}{L+W} \qquad \text{eq. 104-1}$$

where:

D_e = equivalent diameter
L = length
W = width

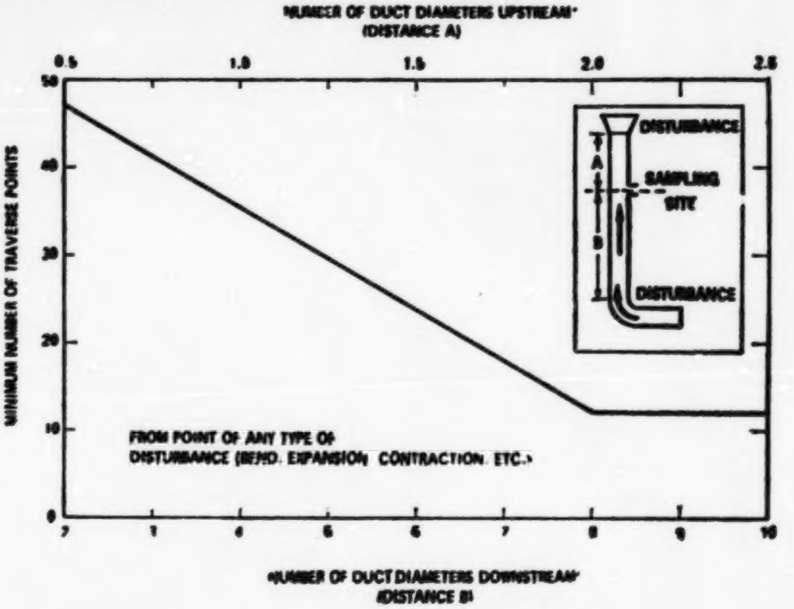

Figure 101-3. Minimum number of traverse points.

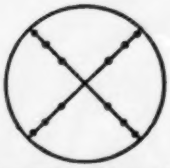

Figure 104-4. Cross section of circular stack showing location of traverse points on perpendicular diameters.

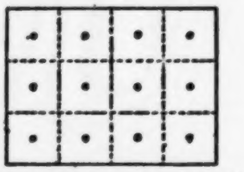

Figure 104-5. Cross section of rectangular stack divided into 12 equal areas, with traverse points at centroid of each area.

4.2.3 When the above sampling site criteria can be met, the minimum number of traverse points is four (4) for stacks 1 foot in diameter or less, eight (8) for stacks larger than 1 foot but 2 feet in diameter or less, and twelve (12) for stacks larger than 2 feet.

4.2.4 Some sampling situations may render the above sampling site criteria impractical. When this is the case, choose a convenient sampling location and use figure 104-3 to determine the minimum number of traverse points. However, use figure 104-3 only for stacks 1 foot in diameter or larger.

4.2.5 To use figure 104-3, first measure the distance from the chosen sampling location to the nearest upstream and downstream disturbances. Divide this distance by the diameter or equivalent diameter to determine the distance in terms of pipe diameters. Determine the corresponding number of traverse points for each distance from figure 104-3. Select the higher of the two numbers of traverse points, or a greater value, such that for circular stacks the number is a multiple of four, and for rectangular stacks the number follows the criteria of section 4.3.2.

4.2.6 If a selected sampling point is closer than 1 inch from the stack wall, adjust the location of that point to ensure that the sample is taken at least 1 inch away from the wall.

4.3 Cross-sectional layout and location of traverse points.

Table 104-1. Location of traverse points in circular stacks
(Percent of stack diameter from inside wall to traverse point)

Traverse point number on a diameter	Number of traverse points on a diameter											
	2	4	6	8	10	12	14	16	18	20	22	24
1	14.6	6.7	4.4	3.3	2.5	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	85.4	25.0	14.7	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3		75.0	29.5	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5
4		93.3	70.5	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9
5			85.3	67.7	34.2	25.0	20.1	16.9	14.6	12.9	11.6	10.5
6			95.6	80.6	65.8	35.5	26.9	22.0	18.8	16.5	14.6	13.2
7				89.5	77.4	64.5	36.6	28.3	23.6	20.4	18.0	16.1
8				96.7	85.4	75.0	63.4	37.5	29.6	25.0	21.8	19.4
9					91.8	82.3	73.1	62.5	38.2	30.6	26.1	23.0
10					97.5	88.2	79.9	71.7	61.8	38.8	31.5	27.2
11						93.3	85.4	78.0	70.4	61.2	39.3	32.3
12						97.9	90.1	83.1	76.4	69.4	60.7	39.8
13							94.3	87.5	81.2	75.0	68.5	60.2
14							98.2	91.5	85.4	79.6	73.9	67.7
15								95.1	89.1	83.5	78.2	72.8
16								98.4	92.5	87.1	82.0	77.0
17									95.6	90.3	85.4	80.6
18									98.6	93.3	88.4	83.9
19										96.1	91.3	86.8
20										98.7	94.0	89.5
21											96.5	92.1
22											98.9	94.5
23												96.8
24												98.9

4.3.1 For circular stacks locate the traverse points on at least two diameters according to figure 104-4 and table 104-1. The traverse axes shall divide the stack cross section into equal parts.

4.3.2 For rectangular stacks divide the cross section into as many equal rectangular areas as traverse points, such that the ratio of the length to the width of the elemental areas is between 1 and 2. Locate the traverse points at the centroid of each equal area according to figure 104-5.

4.4 *Measurement of stack conditions.*—4.4.1 Set up the apparatus as shown in figure 104-2. Make sure all connections are tight and leak free. Measure the velocity head and temperature at the traverse points specified by §§ 4.2 and 4.3.

4.4.2 Measure the static pressure in the stack.

4.4.3 Determine the stack gas moisture.

4.4.4 Determine the stack gas molecular weight from the measured moisture content and knowledge of the expected gas stream composition. A standard Orsat analyzer has been found valuable at combustion sources. In all cases, sound engineering judgment should be used.

4.5 *Preparation of sampling train.*—4.5.1 Prior to assembly, clean all glassware (probe, impingers, and connectors) by soaking in wash acid for 2 hours. Place 100 ml of distilled water in each of the first two impingers, leave the third impinger empty, and place approximately 200 g of preweighted silica gel in the fourth impinger. Save a portion of the distilled water as a blank in the sample analysis. Set up the train and the probe as in figure 104-1.

4.5.2 Leak check the sampling train at the sampling site. The leakage rate should not be in excess of 1 percent of the desired sampling rate. If condensation in the probe or filter is a problem, probe and filter heaters will be required. Adjust the heaters to provide a temperature at or above the stack temperature. However, membrane filters such as the Millipore AA are limited to about 225° F. If the stack gas is in excess of about 200° F., consideration should be given to an alternate procedure such as moving the filter holder downstream of the first impinger to insure that the filter does not exceed its temperature limit. Place crushed ice around the impingers. Add more ice during the test to keep the temperature of the gases leaving the last impinger at 70° F. or less.

4.6 *Beryllium train operation.*—4.6.1 For each run, record the data required on the example sheet shown in figure 104-6. Take readings at each sampling point at least every 5 minutes and when significant changes in stack conditions necessitate additional adjustments in flow rate.

4.6.2 Sample at a rate of 0.5 to 1.0 ft.³/min. Samples shall be taken over such a period or periods as are necessary to accurately determine the maximum emissions which would occur in a 24-hour period. In the case of cyclic operations, sufficient tests shall be made so as to allow accurate determination or calculation of the emissions which will occur over the duration of the cycle. A minimum sample time of 2 hours is recommended.

PLANT________
LOCATION________
OPERATOR________
DATE________
RUN NO.________
SAMPLE BOX NO.________
METER BOX NO.________
METER $\Delta H_@$________
C FACTOR________

SCHEMATIC OF STACK CROSS SECTION

AMBIENT TEMPERATURE________
BAROMETRIC PRESSURE________
ASSUMED MOISTURE, %________
HEATER BOX SETTING________
PROBE LENGTH, m.,________
NOZZLE DIAMETER, in.________
PROBE HEATER SETTING________

TRAVERSE POINT NUMBER	SAMPLING TIME (θ), min.	STATIC PRESSURE (P_s), in. Hg.	STACK TEMPERATURE (T_s), °F	VELOCITY HEAD (ΔP_s).	PRESSURE-DIFFERENTIAL ACROSS ORIFICE METER (ΔH), in. H_2O	GAS SAMPLE VOLUME (V_m), ft³	GAS SAMPLE TEMPERATURE AT DRY GAS METER		SAMPLE BOX TEMPERATURE, °F	IMPINGER TEMPERATURE, °F
							INLET ($T_{m_{in}}$), °F	OUTLET ($T_{m_{out}}$), °F		
TOTAL							Avg.	Avg.		
AVERAGE							Avg.			

Figure 104-6. Field data

4.6.3 To begin sampling, position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Sample for at least 5 minutes at each traverse point; sampling time must be the same for each point. Maintain isokinetic sampling throughout the sampling period. Nomographs which aid in the rapid adjustment of the sampling rate without other computations are in APTD-0576 and are available from commercial suppliers. Note that standard monographs are applicable only for type S pitot tubes and air or a stack gas with an equivalent density. Contact EPA or the sampling train supplier for instructions when the standard monograph is not applicable.

4.6.4 Turn off the pump at the conclusion of each run and record the final readings. Immediately remove the probe and nozzle from the stack and handle in accordance with the sample recovery process described in § 4.7.

4.7 *Sample recovery.*—4.7.1 (All glass storage bottles and the graduated cylinder must be precleaned as in § 4.5.1.) This operation should be performed in an area free of possible beryllium contamination. When the sampling train is moved, care must be exercised to prevent breakage and contamination.

4.7.2 Disconnect the probe from the impinger train. Remove the filter and any loose particulate matter from the filter holder and place in a sample bottle. Place the contents (measured to ±1 ml) of the first three impingers into another sample bottle. Rinse the probe and all glassware between it and the back half of the third impinger with water and acetone, and add this to the latter sample bottle. Clean the probe with a brush or a long slender rod and cotton balls. Use acetone while cleaning. Add these to the sample bottle. Retain a sample of the water and acetone as a blank. The total amount of wash water and acetone used should be measured for accurate blank correction. Place the silica gel in the plastic jar. Seal and secure all sample containers for shipment. If an additional test is desired, the glassware can be carefully double rinsed with distilled water and reassembled. However, if the glassware is to be out of use more than 2 days, the initial acid wash procedure must be followed.

4.8 Analysis.

4.8.1 *Apparatus preparation.*—Clean all glassware according to the procedure of section 4.5.1. Adjust the instrument settings according to the instrument manual, using an absorption wavelength of 234.8 nm.

4.8.2 *Sample preparation.*—The digestion of beryllium samples is accomplished in part in concentrated perchloric acid. *Caution:* The analyst must insure that the sample is heated to light brown fumes after the initial nitric acid addition; otherwise, dangerous perchlorates may result from the subsequent perchloric acid digestion. Perchloric acid also should be used only under a perchloric acid hood.

4.8.2.1 Transfer the filter and any loose particulate matter from the sample container to a 150 ml beaker. Add 35 ml concentrated nitric acid. Heat on a hotplate until light brown fumes are evident to destroy all organic matter. Cool to room temperature and add 5 ml concentrated sulfuric acid and 5 ml concentrated perchloric acid. Then proceed with step 4.8.2.4.

4.8.2.2 Place a portion of the water and acetone sample into a 150 ml beaker and put on a hotplate. Add portions of the remainder as evaporation proceeds and evaporate to dryness. Cool the residue and add 35 ml concentrated nitric acid. Heat on a hotplate until light brown fumes are evident to destroy any organic matter. Cool to room temperature and add 5 ml concentrated sulfuric acid, and 5 ml concentrated perchloric acid. Then proceed with step 4.8.2.4.

4.8.2.3 Weigh the spent silica gel and report to the nearest gram.

4.8.2.4 Samples from 4.8.2.1 and 4.8.2.2 may be combined here for ease of analysis. Replace on a hotplate and evaporate to dryness in a perchloric acid hood. Cool and dissolve the residue in 10.0 ml of 25 percent V/V hydrochloric acid. Samples are now ready for the atomic absorption unit. The beryllium concentration of the sample must be within the calibration range of the unit. If necessary, further dilution of sample with 25 percent V/V hydrochloric acid must be performed to bring the sample within the calibration range.

4.8.3 *Beryllium determination.*—Analyze the samples prepared in 4.8.2 at 234.8 nm using a nitrous oxide/acetylene flame. Aluminum, silicon and other elements can interfere with this method if present in large quantities. Standard methods are available, however, to effectively eliminate these interferences (see Reference 5).

5. *Calibration*—5.1 *Sampling train.*—5.1.1 Use standard methods and equipment as detailed in APTD-0576 to calibrate the rate meter, pitot tube, dry gas meter and probe heater (if used). Recalibrate prior to each test series.

5.2 *Analysis.*—5.2.1 Standardization is made with the procedure as suggested by the manufacturer with standard beryllium solution. Standard solutions will be prepared from the stock solution by dilution with 25 percent V/V hydrochloric acid. The linearity of working range should be established with a series of standard solutions. If collected samples are out of the linear range, the samples should be diluted. Standards should be interspersed with the samples since the calibration can change slightly with time.

6. *Calculations*—6.1 *Average dry gas meter temperature, stack temperature, stack pressure and average orifice pressure drop.*—See data sheet (figure 104-6).

6.2 *Dry gas volume.*—Correct the sample volume measured by the dry gas meter to stack conditions by using equation 104-2.

$$V_{m_s} = V_m \frac{T_s}{T_m} \frac{\left(P_{bar} + \frac{\Delta H}{13.6}\right)}{P_s} \qquad \text{eq. 104-2}$$

where:

V_{m_s} = Volume of gas sample through the dry gas meter (stack conditions), ft³.

V_m = Volume of gas sample through the dry gas meter (meter conditions), ft³.

T_s = Average temperature of stack gas, °R.

T_m = Average dry gas meter temperature, °R.

P_{bar} = Barometric pressure at the orifice meter, in Hg.

ΔH = Average pressure drop across the orifice meter, in H_2O.

13.6 = Specific gravity of mercury.

P_s = Stack pressure, P_{bar} ± static pressure, in Hg.

6.3 *Volume of water vapor.*

$$W_{w_s} = K_w V_{l_c} \frac{T_s}{P_s} \qquad \text{eq. 104-3}$$

where:

V_{w_s} = Volume of water vapor in the gas sample (stack conditions), ft³.

$K_w = 0.00267 \frac{\text{in Hg-ft}^3}{\text{ml-°R}}$, when these units are used.

V_{l_c} = Total volume of liquid collected in impingers and silica gel (see figure 104-7), ml.

T_s = Average stack gas temperature, °R.

P_s = Stack pressure, P_{bar} ± static pressure, in Hg.

6.4 *Total gas volume.*

$$V_{total} = V_{m_s} + V_{w_s} \qquad \text{eq. 104-4}$$

where:

V_{total} = Total volume of gas sample (stack conditions), ft³.

V_{m_s} = Volume of gas through dry gas meter (stack conditions), ft³.

V_{w_s} = Volume of water vapor in gas sample (stack conditions), ft³.

6.5 *Stack gas velocity.*

Use equation 104-5 to calculate the stack gas velocity.

$$(v_s)_{avg.} = K_p C_p (\sqrt{\Delta p})_{avg.} \sqrt{\frac{(T_s)_{avg.}}{P_s M_s}} \qquad \text{eq. 104-5}$$

where:

$(v_s)_{avg.}$ = Average stack gas velocity, feet per second.

$K_p = 85.53 \frac{\text{ft}}{\text{sec}} \left(\frac{\text{lb-in Hg}}{\text{lb mole-°R-in } H_2O}\right)^{1/2}$, when these units are used.

C_p = Pitot tube coefficient, dimensionless.

$(T_s)_{avg.}$ = Average stack gas temperature, °R.

$(\sqrt{\Delta p})_{avg.}$ = Average square root of the velocity head of stack gas $(\text{in } H_2O)^{1/2}$ (see figure 104-6).

P_s = Stack pressure, P_{bar} ± static pressure, in Hg.

M_s = Molecular weight of stack gas (wet basis)—the summation of the products of the molecular weight of each component multiplied by its volumetric proportion in the mixture, lb/lb-mole.

	VOLUME OF LIQUID WATER COLLECTED	
	IMPINGER VOLUME, ml	SILICA GEL WEIGHT, g
FINAL		
INITIAL		
LIQUID COLLECTED		
TOTAL VOLUME COLLECTED		g* ml

*CONVERT WEIGHT OF WATER TO VOLUME BY dividing total weight INCREASE BY DENSITY OF WATER. (1 g/ml):

$$\frac{\text{INCREASE, g}}{(1 \text{ g/ml})} = \text{VOLUME WATER, ml}$$

Figure 104-7. Analytical data.

PLANT______________________________

DATE______________________________

RUN NO.___________________________

STACK DIAMETER, in.__________________

BAROMETRIC PRESSURE, in. Hg._____________

STATIC PRESSURE IN STACK (P_g), in. Hg.________

OPERATORS__________________________

SCHEMATIC OF STACK CROSS SECTION

Traverse point number	Velocity head, in. H_2O	$\sqrt{\Delta_p}$	Stack Temperature (T_S), °F
	AVERAGE:		

Figure 104-8. Velocity traverse data.

Figure 104-8 shows a sample recording sheet for velocity traverse data. Use the averages in the last two columns of figure 104-8 to determine the average stack gas velocity from equation 104-5.

6.6 *Beryllium collected.*—Calculate the total weight of beryllium collected by using equation 104-6.

$$W_t = V_tC_t - V_wC_w - V_aC_a \quad \text{eq. 104-6}$$

where:

W_t = Total weight of beryllium collected, μg.

V_t = Total volume of hydrochloric acid from step 4.8.2.4, ml.

C_t = Concentration of beryllium found in sample, μg/ml.

V_w = Total volume of water used in sampling (impinger contents plus all wash amounts), ml.

C_w = Blank concentration of beryllium in water, μg/ml.

V_a = Total volume of acetone used in sampling (all wash amounts), ml.

C_a = Blank concentration of beryllium in acetone, μg/ml.

6.7 *Total beryllium emissions.*—Calculate the total amount of beryllium emitted from each stack per day by equation 104-7. This equation is applicable for continuous operations. For cyclic operations, use only the time per day each stack is in operation. The total beryllium emissions from a source will be the summation of results from all stacks.

$$R = \frac{W_t (v_s)_{avg.} A_s}{V_{total}} \times \frac{86{,}400 \text{ seconds/day}}{10^6 \ \mu g/g}$$

eq. 104-7

where:

R = Rate of emission, g/day.

W_t = Total weight of beryllium collected, μg.

V_{total} = Total volume of gas sample (stack conditions), ft³.

$(v_s)_{avg.}$ = Average stack gas velocity, feet per second.

A_s = Stack area, ft².

6.8 *Isokinetic variation (comparison of velocity of gas in probe tip to stack velocity).*

$$I = \frac{100 V_{total}}{A_n \Theta (vs)_{avg.}}$$

eq. 104-8

where:

I = Percent of isokinetic sampling.

V_{total} = Total volume of gas sample (stack conditions), ft³.

A_n = Probe tip area, ft².

Θ = Sampling time, sec.

$(v_s)_{avg.}$ = Average stack gas velocity, feet per second.

7. *Evaluation of results*—7.1 *Determination of compliance.*—7.1.1 Each performance test shall consist of three repetitions of the applicable test method. For the purpose of determining compliance with an applicable national emission standard, the average of results of all repetitions shall apply.

7.2 *Acceptable isokinetic results.*—7.2.1 The following range sets the limit on acceptable isokinetic sampling results:

If 90 percent ≤ I ≤ 110 percent, the results are acceptable; otherwise, reject the test and repeat.

7. *References.*—1. Addendum to Specifications for Incinerator Testing at Federal Facilities, PHS, NCAPC, December 6, 1967.

2. Amos, M. D., and Willis, J. B., "Use of High-Temperature Pre-Mixed Flames in Atomic Absorption Spectroscopy," Spectrochim. Acta, 22: 1325, 1966.

3. Determining Dust Concentration in a Gas Stream, ASME Performance Test Code No. 27, New York, N.Y., 1957.

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5. Fleet, B., Liberty, K. V., and West, T. S., "A Study of Some Matrix Effects in the Determination of Beryllium by Atomic Absorption Spectroscopy in the Nitrous Oxide-Acetylene Flame," Talanta, 17: 203, 1970.

6. Mark, L. S., Mechanical Engineers' Handbook, McGraw-Hill Book Co., Inc., New York, N.Y., 1951.

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16. Standard Method for Sampling Stacks for Particulate Matter, In: 1971 Book of ASTM standards, Part 23, Philadelphia, 1971, ASTM Designation D-2928-71.

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APPENDIX B ILLUSTRATED USE OF NOMOGRAPHS

The material in this appendix is, in the most part, reproduced from APTD-0576 (ref. 5).

NOMOGRAPHS

The correction factor nomograph (fig. B1) and the operating nomograph (fig. B2) have been designed for use with the sampling train as aids for rapid isokinetic sampling rate adjustments and for selection of proper nozzle size. To determine the correction factor, C, on the nomograph, the following information is first required:

(1) Percent moisture, $\%H_2O$. This may be determined from a previous test or presurvey, or before the sample run.

(2) Orifice calibration factor, $\Delta H@$. This is determined from the laboratory calibration (see section on Calibration).

(3) Meter temperature, T_m. Temperature at the meter rises above ambient temperature because of the pump and can easily be estimated with experience. An estimate within 10°F (approximately ± 1 percent error) is all that is necessary (an initial estimate of about 25°F above ambient temperature has been used). This approximation above ambient temperature is not required if the pump is located outside the meter box console.

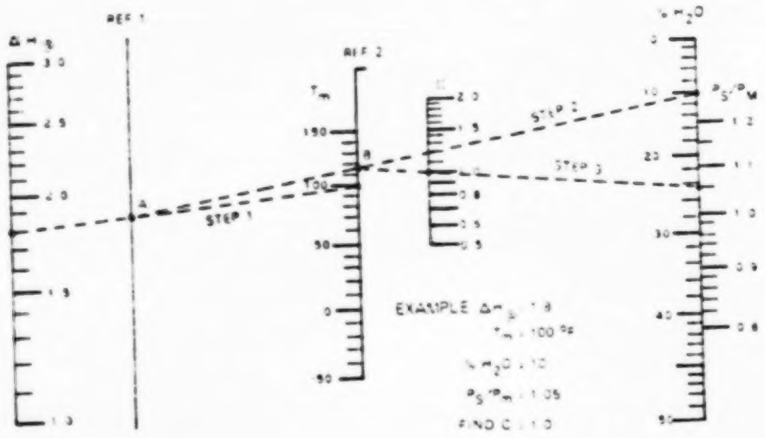
ΔH@
3.0
2.5
2.0
1.5
1.0
REF 1
A
STEP 1
REF 2
T_m
150
B
100
50
0
-50
C
2.0
1.5
1.0
0.8
0.6
0.5
STEP 2
STEP 3
% H_2O
0
10
20
30
40
50
P_S/P_M
1.2
1.1
1.0
0.9
0.8
EXAMPLE ΔH@ = 1.8
T_m = 100 °F
% H_2O = 10
P_S/P_m = 1.05
FIND C = 1.0

Figure B1. Nomograph for correction factor, C.

Preceding page blank

(4) Stack pressure, P_s. This is measured before the sample run; or if the sampling site is near the exit of the stack, atmospheric pressure is used.

(5) Meter pressure, P_m. Same as atmospheric pressure.

To obtain correction-factor, C (fig. B1):

(1) Draw line from $\Delta H@$ to T_m to obtain point "A" on reference line 1 (ref. 1).

(2) Draw line from point "A" to $\%H_2O$ to obtain point "B" on reference line 2 (ref. 2).

(3) Draw line from point "B" to the calculated value P_s/P_m to obtain correction factor, C.

To select the nozzle size and to set the K-factor on the operating nomograph, the following information is first required:

(1) C factor. This is obtained from the correction-factor nomograph (fig. B1).

> Note: If the coefficient, C_p, of the type-S pitot tube is not equal to .85 ± .02, the following is required: (a) Multiply C times $\left(\frac{C_p}{.85}\right)$ = C' for the correct C factor in obtaining the K-factor, or (b) if C' is less than 0.5, then use C and multiply each ΔP reading by $\left(\frac{C_p}{.85}\right)^2$ for each adjustment.

(2) Stack temperature, T_s. This is determined in °F by a rough temperature traverse to within ± 25°F before the sample run.

(3) Average velocity pressure, ΔP. This is determined by a rough preliminary pitot traverse, using the average of minimum and maximum ΔP's in inches of water.

(4) Exact available nozzle sizes, D. This is obtained from the calibration of available nozzles.

To select the nozzle size and to set the K-factor pivot point, use the following procedure (fig. B2):

(1) Set correction factor, C, on sliding scale to the reference mark, "A."

(2) Align T_s with average ΔP, note probe tip diameter on D-scale, and select exact nozzle size closest to it.

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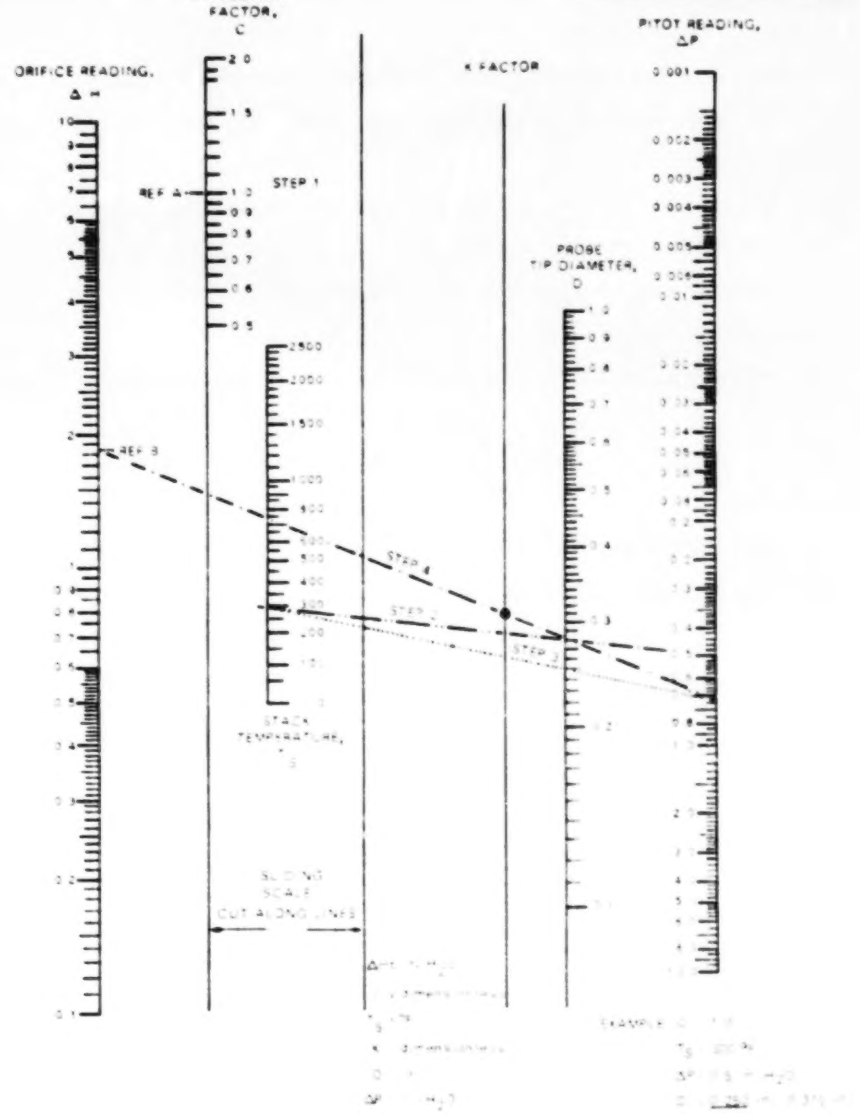

ORIFICE READING, Δ H
CORRECTION FACTOR, C
STACK TEMPERATURE, T_S
K FACTOR
PROBE TIP DIAMETER, D
PITOT READING, ΔP
REF A
REF B
STEP 1
STEP 2
STEP 3
STEP 4
SLIDING SCALE CUT ALONG LINES
EXAMPLE

Figure B2. Use of the nomograph in selecting nozzle size and setting K factor.

(3) Align T_s with exact nozzle size selected and obtain a value on the ΔP scale.

(4) Align the ΔP value with reference mark, "B", on ΔH scale, and set the K-factor pivot point.

To obtain the orifice meter settings, ΔH, for isokinetic conditions after the K-factor pivot point has been set, use the following procedure (fig. B3):

(1) Position the pitobe nozzle at the sampling point.

(2) Read the pitot tube ΔP.

(3) Align the ΔP through the K-factor pivot point

(4) Obtain ΔH and adjust metering valves.

The nomograph assumes the following, once the K-factor pivot point is set:

(1) T_s does not change more than 25° for $T_s < 1000°F$ or 50° for $T_s > 1000°F$.

(2) D is not changed during the test.

(3) T_m was estimated correctly and does not vary more than 10°.

(4) Percent H_2O remains constant, within ± 1.0 percent.

(5) P_s and P_m remain constant, within ± 1.0 percent.

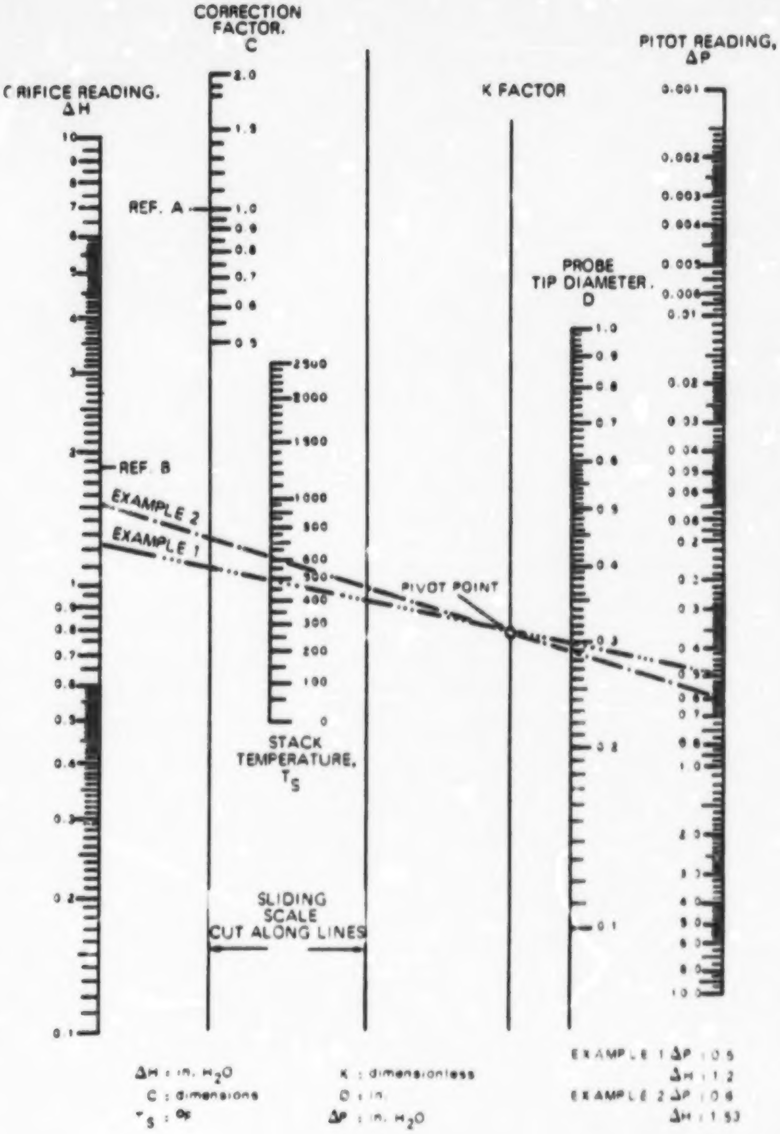

CORRECTION FACTOR, C
PITOT READING, ΔP
ORIFICE READING, ΔH
K FACTOR
PROBE TIP DIAMETER, D
REF. A
REF. B
EXAMPLE 2
EXAMPLE 1
PIVOT POINT
STACK TEMPERATURE, T_S
SLIDING SCALE CUT ALONG LINES
ΔH : in. H_2O
C : dimensions
T_S : °F
K : dimensionless
D : in.
ΔP : in. H_2O
EXAMPLE 1 ΔP : 0.5
ΔH : 1.2
EXAMPLE 2 ΔP : 0.6
ΔH : 1.53

Figure B3. Nomograph operation to obtain desired orifice meter settings.

APPENDIX C ILLUSTRATED AUDIT PROCEDURES

A flow chart of the operations involved in an auditing program from first setting desired limits on the data quality to filing the results is given below. Assumed numbers are used and a sample calculation of an audit is performed in the flow chart. Each operation is referred to the section in the text of the report where it is discussed.

MANAGER

1. LIMITS FOR DATA QUALITY CAN BE SET BY WHAT IS DESIRED OR FROM THE NATURAL VARIABILITY OF THE METHOD WHEN USED BY TRAINED AND COMPETENT PERSONNEL. FOR THIS EXAMPLE, IT IS ASSUMED THAT CV{$\overline{R}$} = 25.7 PERCENT (subsec.4.1), AND USING $\pm$ 3 CV{$\overline{R}$}, THE LIMITS ARE L = -77.1 PERCENT AND U = 77.1 PERCENT.

2. FROM PRIOR KNOWLEDGE OF DATA QUALITY, ESTIMATE THE PERCENTAGE OF FIELD MEASUREMENTS FALLING OUTSIDE THE ABOVE LIMITS. IF NO INFORMATION IS AVAILABLE, MAKE AN EDUCATED GUESS. IT IS ASSUMED IN THIS EXAMPLE THAT 50 PERCENT OF THE FIELD DATA ARE OUTSIDE THE LIMITS L AND U (subsec. 4.4.4).

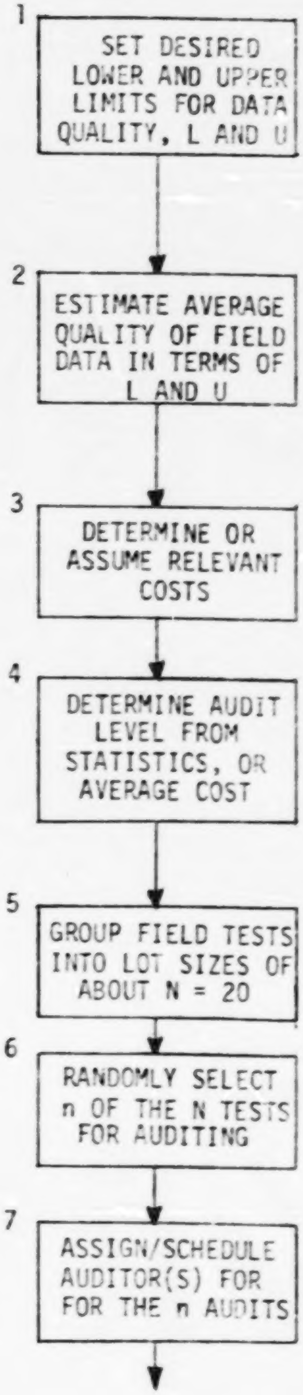
3. DETERMINE: (1) COST OF CONDUCTING AN AUDIT, (2) COST OF FALSELY INFERRING THAT GOOD DATA ARE BAD, (3) COST OF FALSELY INFERRING THAT BAD DATA ARE GOOD, AND (4) COST SAVINGS FOR CORRECTLY IDENTIFYING BAD DATA (subsec. 4.4.4).

4. DETERMINE THE AUDIT LEVEL EITHER BY (1) MINIMIZING AVERAGE COST USING EQUATION (17) OF SUBSECTION 4.4.4, OR (2) ASSURING A DESIRED LEVEL OF CONFIDENCE IN THE REPORTED DATA THROUGH STATISTICS. FOR THIS EXAMPLE, THE AUDIT LEVEL IS TAKEN AS n = 5 (fig. 11).

5. BY TEAMS, TYPES OF SOURCES, OR GEOGRAPHY, GROUP FIELD TESTS INTO LOTS (GROUPS) OF ABOUT 20 THAT WILL BE PERFORMED IN A PERIOD OF ONE CALENDAR QUARTER.

6. SELECT n OF THE N TESTS FOR AUDITING. COMPLETE RANDOMIZATION MAY NOT BE POSSIBLE DUE TO AUDITOR'S SCHEDULE. THE PRIMARY POINT IS THAT THE FIELD TEAM SHOULD NOT KNOW IN ADVANCE THAT THEIR TEST IS TO BE AUDITED.

7. ASSIGN OR SCHEDULE AN AUDITOR FOR EACH FIELD TEST.


1
SET DESIRED LOWER AND UPPER LIMITS FOR DATA QUALITY, L AND U
2
ESTIMATE AVERAGE QUALITY OF FIELD DATA IN TERMS OF L AND U
3
DETERMINE OR ASSUME RELEVANT COSTS
4
DETERMINE AUDIT LEVEL FROM STATISTICS, OR AVERAGE COST
5
GROUP FIELD TESTS INTO LOT SIZES OF ABOUT N = 20
6
RANDOMLY SELECT n OF THE N TESTS FOR AUDITING
7
ASSIGN/SCHEDULE AUDITOR(S) FOR FOR THE n AUDITS

AUDITOR

8. THE AUDITOR OBTAINS APPROPRIATE CALIBRATED EQUIPMENT AND SUPPLIES FOR THE AUDIT (subsec. 4.3).

9. OBSERVE THE FIELD TEAM'S PERFORMANCE OF THE FIELD TEST. FILL IN THE AUDITOR'S CHECKLIST (table 4) AND NOTE ANY UNUSUAL CONDITIONS THAT OCCURRED DURING THE TEST.

10. IN THE FIELD TEAM'S HOME LABORATORY, MAKE INDEPENDENT DETERMINATIONS OF C_p, D_n, γ, AND $\Delta H@$ (subsec. 4.3) ACCORDING TO THE CALIBRATION PROCEDURES GIVEN IN SUBSECTION 2.2.

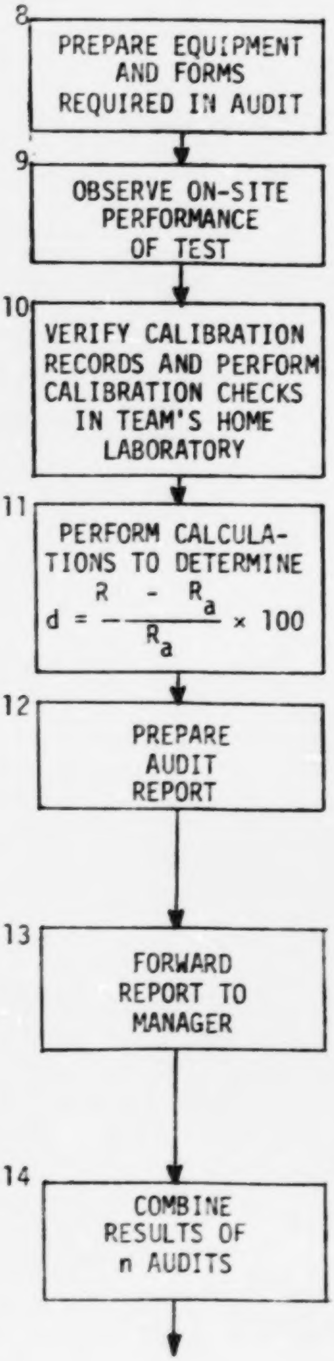
11. STARTING WITH THE RAW DATA FROM THE FIELD AND USING AUDIT VALUES, PERFORM ALL THE CALCULATIONS NECESSARY TO ARRIVE AT A VALUE FOR d (subsec. 4.3, fig. 13).

12. THE AUDITOR'S REPORT SHOULD INCLUDE (1) DATA SHEET FILLED OUT BY THE FIELD TEAM (fig. 5), (2) AUDITOR'S CHECKLIST WITH COMMENTS (table 4), (3) AUDIT DATA SHEET WITH CALCULATIONS (fig. 13), and (4) A SUMMARY OF THE TEAM'S PERFORMANCE WITH A NUMERICAL RATING (subsec. 4.3).

13. THE AUDITOR'S REPORT IS FORWARDED TO THE MANAGER.

MANAGER

14. COLLECT THE AUDITOR'S REPORTS FROM THE n AUDITS OF THE LOT OF N STACKS. IN THIS CASE n = 5 AND ASSUMED VALUES FOR THE AUDITS ARE d_1 = 32.0, d_2 = 19.5, d_3 = 10.3, d = 12.1, and d_5 = 14.6 (table 5).


8
PREPARE EQUIPMENT AND FORMS REQUIRED IN AUDIT
9
OBSERVE ON-SITE PERFORMANCE OF TEST
10
VERIFY CALIBRATION RECORDS AND PERFORM CALIBRATION CHECKS IN TEAM'S HOME LABORATORY
11
PERFORM CALCULATIONS TO DETERMINE
$d = \frac{R - R_a}{R_a} \times 100$
12
PREPARE AUDIT REPORT
13
FORWARD REPORT TO MANAGER
14
COMBINE RESULTS OF n AUDITS

15. CALCULATE $\bar{d}$ AND s_d ACCORDING TO THE SAMPLE IN TABLE 10. RESULTS OF THIS SAMPLE CALCULATION SHOW $\bar{d}$ = 17.7%, AND s_d = 8.7% (table 5, subsec. 4.4.3).

16. USE A t-TEST TO CHECK $\bar{d}$ FOR SIGNIFICANCE, FOR THIS EXAMPLE $t = 17.7\sqrt{5}/8.7 = 4.5$. THE TABULATED t-VALUE FOR 4 DEGREES OF FREEDOM AT THE 0.05 LEVEL IS 2.132; HENCE, $\bar{d}$ IS NOT SIGNIFICANTLY DIFFERENT FROM 0 AT THIS LEVEL. ALSO, s_d IS CHECKED AGAINST THE ASSUMED VALUE OF 25.7 PERCENT BY A CHI-SQUARE TEST. $\chi^2/f = s_d^2/\sigma^2\{\bar{d}\} = (8.7)^2/(25.7)^2 = 0.11$, THE TABULATED VALUE OF $\chi^2/4$ AT THE 95 PERCENT LEVEL IS 0.711; HENCE, s_d IS NOT SIGNIFICANTLY DIFFERENT FROM 25.7 PERCENT.

17. OBTAIN THE VALUE OF k FROM TABLE 11, FOR n = 5 AND p = 0.1. THIS VALUE IS 2.742, THEN $\bar{d} + k\ s_d = 17.7 + (1.976)(8.7) = 34.9$ AND $\bar{d} - k\ s_d = 17.7 + (1.976)(8.7) = .51$ (subsec. 4.4.3).

18. COMPARE THE ABOVE CALCULATIONS WITH LIMITS L AND U (subsec. 4.4.3). FOR THIS EXAMPLE

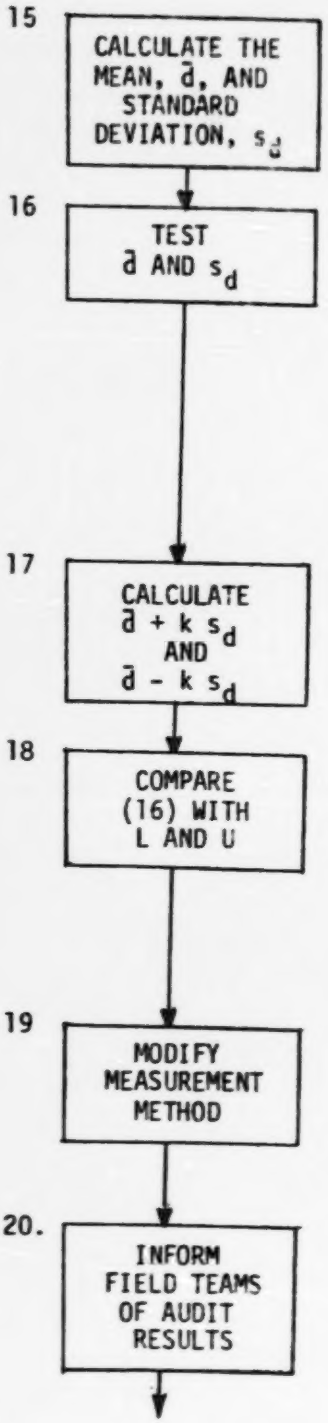
$$\bar{d} + k\ s_d = 41.6 < U = 77.1$$

$$\bar{d} - k\ s_d = -6.2 > L = -77.1$$

BOTH CONDITIONS ARE SATISFIED, GO TO 20. (IF EITHER OF THE LIMITS HAD BEEN EXCEEDED, CONTINUE TO 19.)

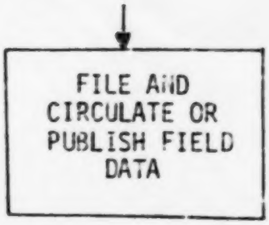
19. STUDY THE AUDIT AND FIELD DATA FOR SPECIFIC AREAS OF VARIABILITY, SELECT THE MOST COST-EFFECTIVE ACTION OPTION(S) THAT WILL RESULT IN GOOD QUALITY DATA (subsec. 4.2). NOTIFY THE FIELD TEAMS TO IMPLEMENT THE SELECTED ACTION OPTION(S).

20. A COPY OF THE AUDITOR'S REPORT SHOULD BE SENT TO THE RESPECTIVE FIELD TEAM. ALSO, THE DATA ASSESSMENT RESULTS, i.e., CALCULATED VALUES OF $\bar{d}$, s_d, AND COMPARISON WITH THE LIMITS L AND U SHOULD BE FORWARDED TO EACH TEAM INVOLVED IN THE N FIELD TESTS.


15
CALCULATE THE MEAN, $\bar{d}$, AND STANDARD DEVIATION, s_d
16
TEST $\bar{d}$ AND s_d
17
CALCULATE $\bar{d} + k\ s_d$ AND $\bar{d} - k\ s_d$
18
COMPARE (16) WITH L AND U
19
MODIFY MEASUREMENT METHOD
20.
INFORM FIELD TEAMS OF AUDIT RESULTS

21. THE FIELD DATA WITH AUDIT RESULTS ATTACHED ARE FILED. THE AUDIT DATA SHOULD REMAIN WITH THE FIELD DATA FOR ANY FUTURE USES.

21


FILE AND CIRCULATE OR PUBLISH FIELD DATA

APPENDIX D GLOSSARY OF SYMBOLS

This is glossary of symbols as used in this document. Symbols used and defined in the reference method (appendix A) are not repeated here.

SYMBOL	DEFINITION
N	Lot size, i.e., the number of field tests to be treated as a group.
n	Sample size for the quality audit (section IV).
CV{X}	Assumed or known coefficient of variation (100 σ_X/μ_X)
CV{X}	Computed coefficient of variation (100 $s_X/\bar{X}$) from a finite sample of measurements.
$\sigma\{X\}$	Assumed standard deviation of the parameter X (population standard deviation).
$s\{X\}$	Computed standard deviation of a finite sample of measurements (sample standard deviation)
$\hat{\tau}\{X\}$	Computed bias of the parameter X for a finite sample (sample basis).
R_g	Range, i.e., the difference in the largest and smallest values in r replicate analyses.
$\varepsilon\{M_n\}$	Random error associated with the measurement of particulate mass, M_n.
d_j	The percent difference in the audit value and the value of $\bar{R}$ arrived at by the field crew for the j^{th} audit.
$\bar{d}$	Mean difference between R_j and R_{aj} for n audits expressed as a percent.
s_d	Computed standard deviation of differences between R_j and R_{aj} expressed as a percent.
p	Percent of measurement outside specified limits L and U.
k	Constant used in sampling by variables (section IV).
P{Y}	Probability of event Y occurring
$t_{(n-1)}$	Statistic used to determine if the sample bias, $\bar{d}$, is significantly different from zero (t-test).
$\chi^2/(n-1)$	Statistic used to determine if the sample variance, s^2, is significantly different from the assumed variance, σ^2, of the parent distribution (chi-square test)

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APPENDIX D GLOSSARY OF SYMBOLS (CONTINUED)

SYMBOL	DEFINITION
L	Lower quality limit used in sampling by variables.
U	Upper quality limit used in sampling by variables.
CL	Center line of a quality control chart.
LCL	Lower control limit of a quality control chart.
UCL	Upper control limit of a quality control chart.
R	Beryllium emission rate reported by the field team for a sample run.
R_a	Beryllium emission rate calculated by the auditor.
$\bar{R}$	Average beryllium emission rate for a field test, i.e., the average of three sample runs, g/d.

APPENDIX E GLOSSARY OF TERMS

The following glossary lists and defines the statistical terms as used in this document.

Accuracy	A measure of the error of a process expressed as a comparison between the average of the measured values and the true or accepted value. It is a function of precision and bias.
Bias	The systematic or nonrandom component of measurement error.
Lot	A specified number of objects to be treated as a group, e.g., the number of field tests to be conducted by an organization during a specified period of time.
Measurement method	A set of procedures for making a measurement.
Measurement process	The process of making a measurement including method, personnel, equipment, and environmental conditions.
Population	The totality of the set of items, units, or measurements, real or conceptual, that is under consideration.
Precision	The degree of variation among successive, independent measurements (e.g., on a homogeneous material) under controlled conditions, and usually expressed as a standard deviation or, as is done here, as a coefficient of variation.
Quality audit	A management tool for independently assessing data quality.
Quality control check	Checks made by the field crew on certain items of equipment and procedures to assure data of good quality.
Sample	Objects drawn, usually at random, from the lot for checking or auditing purposes.

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APPENDIX F CONVERSION FACTORS

Conversion factors for converting the U.S. customary units to the International System of Units (SI)* are given below.

To Convert from	To	Multiply by
	Length	
foot	meter (m)	0.3048
inch	meter (m)	0.0254
	Pressure	
inch of mercury (in. of Hg) (32°F)	Newton/meter^2 (N/m^2)	3386.389
inch of mercury (in. of Hg) (60°F)	Newton/meter^2 (N/m^2)	3376.85
millimeter mercury (mmHg) (32°F)	Newton/meter^2 (N/m^2)	133.3224
inch of water (in. of H_2O) (29.2°F)	Newton/meter^2 (N/m^2)	249.082
inch of water (in. of H_2O) (60°F)	Newton/meter^2 (N/m^2)	248.84
	Force	
pound-force (lbf avoirdupois)	Newton (N)	4.448222
	Mass	
pound-mass (lbm avoirdupois)	kilogram (kg)	0.4535924
	Temperature	
degree Celsius	kelvin (K)	$t_K = t_C + 273.15$
degree fahrenheit	kelvin (K)	$t_K = (t_F + 459.67)/1.8$
degree rankine	kelvin (K)	$t_K = t_R/1.8$
degree fahrenheit	degree Celsius	$t_C = (t_F - 32)/1.8$
kelvin	degree Celsius	$t_C = t_K - 273.15$
	Velocity	
foot/second	meter/second (m/s)	0.3048
foot/minute	meter/second (m/s)	0.00508
	Volume	
cubic foot (ft^3)	meter^3 (m^3)	0.02832
	Volume/Time	
foot^3/minute	meter^3/second (m^3/s)	0.0004719
foot^3/second	meter^3/second (m^3/s)	0.02832

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*Metric Practice Guide (A Guide to the Use of SI, the International Systems of Units), American National Standard Z210.1-1971, American Society for Testing and Materials, ASTM Designation:E380-70, Philadelphia, Pa., 1971.